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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

1598. **Bibliography of publications dealing with the polarographic method in 1957.** J. Heyrovský. *Coll. Czech. Chem. Commun.*, 1958, **23**, Suppl. 1.—This bibliography contains 945 references to publications in 1957 together with 162 references omitted from the bibliographies published in previous years. There is also a list of 151 theses and communications at congresses not fully published.

1599. **Advances in the use of organic reagents in inorganic analysis in the USSR during the last forty years.** V. I. Shlenskaya (Anal. Chem. Dept., Moscow Univ.). *Vestn. Moskov. Univ.*, 1957, (6), 237–250.—A review with 135 references.

G. S. SMITH

1600. **Separation procedure for cations based on paper chromatography.** E. Blasius and W. Göttling (Anorg. Chem. Inst., Tech. Univ., Berlin). *Z. anal. Chem.*, 1958, **162** (6), 423–429.—After the separation of the cations into the 10 groups as described by Jander and Wendt (*Lehrbuch der anal. und präp. Chemie*, Verlag Hörzel, Stuttgart, 1954), a scheme that takes into account some 37 cations, the ppt. are dissolved in HCl or HCl plus H_2O_2 and 3-μl samples are chromatographed on filter-paper according to the "wick-circular" technique (Giri and Rao, *Brit. Abstr. C*, 1952, 556). A large number of mixtures of cations have been separated successfully by this procedure, even when one component is present in a considerable excess. The chromatography of a comparison soln. next to the test soln. is recommended. J. H. WATON

1601. **Triethanolamine as a reagent in qualitative analysis.** I. P. Ryazanov and V. D. Chistota. *Sb. Nauch. Trud. Magnitogorsk. Gorno-Metallurg. Inst.*, 1958, (16), 146–153; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,489.—The use of triethanolamine (**I**) as a precipitant for the separation of mixtures of cations is studied. On adding a 20% soln. of **I** to soln. containing Al^{3+}, Mn^{2+}, Zn^{2+}, Bi^{3+}, Sn^{2+}, Sb^{3+}, Sb^{5+} and Fe^{3+}, ppt. are formed, insol. in excess of **I**. With **I**, Fe^{3+}, Cr^{3+}, Cu^{2+}, Co^{2+} and Ni^{2+} form coloured soluble complexes, while Hg_2^{2+}, Hg^{2+}, Pb^{2+} and Sn^{4+} form colourless complexes. It is established that, in combination with certain other precipitants (oxalate, SCN^-, I^- and $Cr_2O_7^{3-}$), **I** is useful for separating a series of chemically similar cations, *e.g.*, Co^{2+} and Ni^{2+}, Hg^{2+} and Hg_2^{2+}, etc. It is also established that the action of EDTA (disodium salt), diethyldithiocarbamate, 8-hydroxyquinoline and anthranilic acid on cations in the presence of **I** differs in many instances from their action in its absence. On the basis of the results obtained, a procedure is evolved for the qual. analysis of cations without hydrogen sulphide separation. C. D. KOPKIN

1602. **Study of some organic compounds of selenium from the viewpoint of their analytical applications.** J. M. Balcells García de la Torre (Laguna Univ., Teneriffe, Canary Isl.). Thesis, Laguna Univ., 1958, 87 pp.—N′-Benzoyl-N-phenylselenourea (**I**) and N′-benzoyl-NN-diethylselenourea (**II**) have been prepared and their analytical applications studied. In general, the selenoureas form coloured complexes with the noble metals, for which the group Se:C(NRH)– appears to be responsible. The insol. compounds obtained with Ag^+, Au^{3+} and Pd^{2+} have been examined, and the most stable is formed from 1 atom of Pd and 1 molecule of **I**. In HNO_3 medium, **I** (in ethanol or CS_2 soln.) forms colours or ppt. with Ag^+, Au^{3+}, Pd^{2+}, Os^{4+}, Hg^+ and Cu^{2+}; **II** behaves similarly, except that a colour is produced with Cu^{2+} only in neutral medium. These reactions are not highly sensitive. The most suitable conditions for the spectrophotometric determination of Pd^{2+} by means of **I** have been found to be extraction of the Pd (32 to 224 μg) at pH 3·2 to 1·2 into 10 ml of an 0·2% soln. of **I** in CS_2 and measurement of the absorption at 480 mμ. The only appreciable interference is by ions of the other noble metals, when present at a similar level. Osmium^{VIII} (10 to 90 μg) may be similarly determined, extracting from an acid medium with the CS_2 soln. of **I** and measuring at 500 mμ, the soln. of the complex being very stable. **II**, which has the advantage of forming more stable ethanolic soln., may be used similarly to **I** as a qual. reagent, but gives anomalous behaviour in the colorimetric determination of Pd. **II** may be used for the colorimetric determination of Cu^{2+} with a soln. of the reagent in ethanol without extraction, or in CS_2 with extraction, but is less sensitive than dithiocarbamate or dithizone. L. A. O'NEILL

1603. **Optimum pH ranges for complete precipitation of the iron and titanium groups of the Periodic Table with tannic acid.** Chozo Yoshimura and Mitsuo Kiboku (Fac. of Engng, Kinki Univ., Fuse, Osaka). *Japan Analyst*, 1958, **7** (7), 445–449.—The pH range for the quant. pptn. of Fe^{II} (4·6 to 7·8), Fe^{III} (4·5 to 10·3), Ni (7·5 to 9·0), Co (8·3 to 9·3), Mn (8·3 to 10·3), Ti (2·0 to 9·5), Zr (2·0 to 10·4), Hf (4·5 to 9·6) and Th (3·8 to 9·1) with tannic acid (**I**) was studied in ammoniacal buffer. In the presence of citric acid or NH_4HF_2, the ranges change markedly—for Ti (5·0 to 8·8 and 8·5 to 9·8, respectively), Zr (6·1 to 10·4 and 7·4 to 11·4), Hf (6·2 to 11·2 and 7·3 to 10·2) and Th (7·9 to 10·1 and 8·2 to 9·4). The separation of Fe from Mn and from Ni is successful without the aid of other reagents; Ti is separated from Zr, Hf and Th by pptg. the last three with **I** in the presence of H_2O_2.

K. SAITO

1604. **The use of hexamine in quantitative analysis.** J. Lange (Inst. f. Glastechnik, Weisswasser). *Chem. Tech., Berlin*, 1958, **10** (7), 420–421.—The use of hexamine as an analytical reagent for Fe is

described, avoiding the formation of a slimy ppt., difficult to collect and entraining other ions, by not allowing the reacting soln. to boil. To the soln. (100 to 150 ml) of Fe^{III} at pH 3 to 4 are added 2 to 3 g of NH_4Cl and 20 ml of 10% hexamine soln. (slightly acid to methyl red), the soln. is heated almost to boiling-point, and then kept on a boiling-water bath for 45 min. with subsequent filtration, etc. J. L. PROSSER

1605. **Starch - iodine as an indicator.** M. Balázsfalvy (née Zergényi), A. Gröbler, É. Fekete (née Pap), Á. Kellner, L. G. Molnár, M. Nagy, E. Novák (née Sugár) and L. Szekeres (Dept. Agric. Chem., Univ., Budapest). *Magyar Kém. Foly.*, 1958, **64** (7–8), 249–250.—Bromine is used for oxidiations in acid or alkaline medium, and the excess is back-titrated in an HCl medium with As_2O_3 soln. in the presence of KI, ICl or KIO_3 and starch. As long as Br is present, IBr, ICl or IO_3^- do not react with As_2O_3, but at the end-point when all the Br has disappeared, the first drop of As_2O_3 reduces the iodine compound to iodine which produces a blue colour with starch. Numerous examples of this method are given, *e.g.*, the determination of ammonium compounds (including those obtained in the Kjeldahl method), nitrites, oxalates, formaldehyde, formic acid, Fe^{II} and $Fe(CN)_6^{4-}$. The same method of end-point detection can be used with hydrazine sulphate as titrant: it reduces BrO_3^- and bromine, but not iodine. In bromate titrations, the reversible indicator is alkaline 0·1 *N* iodine (1 drop) and starch soln. It becomes colourless at the end-point, when IBr is formed. Suggested uses include the determination of As^{III}, Sb^{III}, $Fe(CN)_6^{4-}$, SCN^- and also ascorbic acid (by using KI and starch). A. G. PETO

1606. **The use of bromine chloride solution in volumetric analysis.** E. Schulek and K. Burger (Inst. for Inorg. and Anal. Chem., Loránd Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1958, **64** (7–8), 241–243.—In the presence of excess of HCl, BrO_3^- and Br^- react to give BrCl (**I**)—

$$BrO_3^- + 2Br^- + 3Cl^- + 6H^+ = 3BrCl + 3H_2O.$$

The soln. of **I** contained $\simeq$ 7% of HCl and its titre changed by only 3 to 5% in several months. This stability is caused by the formation of a chloride complex of **I**, which has a u.v. absorption max. at 343 mμ; its stability const. is $(2{\cdot}6 \pm 1{\cdot}0) \times 10^2$. It is likely that from interhalogen compounds, formed by addition in aq. soln., the halogens separate as halides in the order of their electron affinity, *i.e.*, from **I**, Cl^- are first obtained. The soln. can be used in two ways. In the first, a 0·1 *N* BrO_3^- soln. containing an equiv. of Br^- according to the above equation is added to the unknown, followed by HCl. The excess of **I** is back-titrated iodimetrically. The number of aromatic compounds which can be brominated could not be increased, but the time of bromination has been shortened to 0·5 min. for *o*- and *p*-nitrophenol, to 1 min. for phenol, *p*-hydroxybenzoic acid, *m*-nitrophenol and *m*-cresol, to 2 min. for salicylic acid and to 3 min. for *p*-cresol. If these times are exceeded, **I** oxidises the brominated products giving high results. Even 1% of salicylic acid can be determined in the presence of acetylsalicylic acid: the latter is neither hydrolysed nor brominated in 2 min. The accuracy of all these determinations is $\pm$ 0·5%. In the other method, a 0·1 *N* soln. of **I** containing $\simeq$ 2 *N* HCl was used for the direct titration of hydrazine and its derivatives (phenylhydrazine, semicarbazide and isoniazid). The indicator was 0·2% *p*-ethoxychrysoidine. The accuracy is high, so that hydrazine sulphate can be used (instead of iodimetry) for standardising **I**. A. G. PETO

1607. **Use of iodine chloride in analytical chemistry. X. Micro-titrations with iodine chloride.** J. Čihalík and J. Voráček (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (7), 1269–1273.—The conditions were studied for the oxidative determination of some inorganic compounds on the micro scale by using potentiometric micro-titration with 0·001 *N* to 0·0001 *N* ICl. The factor of the volumetric soln. was determined with standard $K_4Fe(CN)_6$ soln. It was found that 0·6 to 150 μg of As^{3+} in 3 to 100 ml and 2 to 400 μg of $Fe(CN)_6^{4-}$ in 3 to 5 ml can be determined in $NaHCO_3$ medium with good accuracy; 0·1 to 270 μg of I^- in 5 ml can be titrated in 0·05 *N* HCl soln. Chlorides do not interfere; bromides in 150-fold excess cause errors. J. ZÝKA

1608. **Iodine monochloride as volumetric reagent. I. Determinations by mercuric chloride method.** Balwant Singh, Guru Prasad Kashyap and S. S. Sahota (Punjab Univ., Hoshiarpur, India). *Z. anal. Chem.*, 1958, **162** (5), 357–360 (in English).—Antimony potassium tartrate, As_2O_3, ascorbic acid, hydrazine sulphate, and the hydrochlorides of phenylhydrazine, aminoguanidine, 4-phenylsemicarbazone, thiosemicarbazide and semicarbazide can be titrated with 0·1 *N* ICl in the presence of 1 to 2·5 *N* HCl and an excess of $HgCl_2$. Mercuric ions bind the I^- formed during the reaction (*cf.* Miller and Furman, *J. Amer. Chem. Soc.*, 1937, **59**, 152), thus assuring a sharp end-point that is detected (faint violet coloration) with $CHCl_3$ as internal indicator. W. J. BAKER

1609. **Volumetric studies in oxidation - reduction reactions. XII. Oxidation with sodium hypochlorite; iodine monochloride method.** Balwant Singh and Sardul Singh (Punjab Univ. College, Hoshiarpur, India). *Anal. Chim. Acta*, 1958, **19** (1), 10–11 (in English).—Alkaline sodium hypochlorite soln. was used as an oxidant to determine arsenous oxide, hydrazine sulphate, ferrous ammonium sulphate, stannous chloride, sodium sulphite, potassium iodide, mercurous chloride, thallous chloride and antimony potassium tartrate by a volumetric method in which iodine chloride was used as a catalyst and pre-oxidiser. Chloroform was used as an indicator, the pink colour during titration due to dissolved iodine being changed to pale yellow at the end-point owing to the formation of iodine chloride. F. J. M.

1610. **Sodium hypochlorite as volumetric reagent. II. Indirect determination of oxidising agents.** Balwant Singh and Sardul Singh (Punjab Univ., Hoshiarpur, India). *Z. anal. Chem.*, 1958, **162** (5), 325–327 (in English).—Potassium chlorate, $KBrO_3$, KIO_4, $K_2Cr_2O_7$, $K_3Fe(CN)_6$, $KMnO_4$, $K_2S_2O_8$, H_2O_2, $Ce(SO_4)_2$ or chloramine T can be determined by adding 0·1 *N* ferrous ethylenediamine sulphate soln. (20 ml) to the sample in 5 to 6 *N* HCl and titrating the excess of reducing agent with 0·1 *N* NaClO; 0·05 *M* ICl is used as catalyst and pre-oxidiser and $CHCl_3$ as indicator (violet during titration changing to straw colour at the end-point). A slight modification is necessary for $KClO_3$ or $K_3Fe(CN)_6$. W. J. BAKER

1611. **Potassium persulphate as a volumetric reagent. I. The iodine monochloride method.** Balwant Singh and Narinder Singh Ahuja (Chem. Dept., Punjab Univ. College, Hoshiarpur). *J. Indian Chem. Soc.*, 1958, **35** (7), 508–510.—Potassium persulphate is used as a volumetric reagent for the direct determination of As_2O_3, Sb K tartrate, $(NH_4)_2SO_4.FeSO_4$, ferrous ethylenediamine sulphate, HgCl, KSCN, hydrazine sulphate and quinol in 6·0 to 7·5 *N* HCl at room temp., with ICl as a catalyst and pre-oxidiser and $CHCl_3$ as indicator. The violet colour due to the iodine liberated during the titration changes to very pale yellow at the end-point. I. JONES

1612. **Titrations with quinol and analogous reducing agents. IX. Stability of standard quinol solutions.** L. Mráz, V. Simon and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (6), 1083–1088.—The stability of 0·1 *N* to 0·001 *N* soln. of quinol in acid, neutral and alkaline soln. has been studied and the influence of light, oxygen, temp. and heavy metals examined. Volumetric soln. of quinol when slightly acid (1 to 3% of H_2SO_4) do not change during several months, which is advantageous for reductimetric titrations; an inert atmosphere need not be used. In neutral soln. the stability decreases (5 to 7 weeks) and the soln. become brown. Alkaline soln. are unstable and dark products are formed. Freshly prepared soln. of quinol, however, even when acid, change their value immediately after preparation (by 0·5 to 1%) and for volumetric purposes should be used after 1 to 2 days. Potentiometric and spectrophotometric titrations with $K_2Cr_2O_7$ and $Ce(SO_4)_2$ soln. were used in this study. J. ZÝKA

1613. **Mechanism and analytical application of oxidations with ferricyanide, catalysed with osmium tetroxide.** F. Solymosi and A. Varga (Inst. for Inorg. and Anal. Chem., Sci. Univ., Szeged, Hungary). *Magyar Kém. Foly.*, 1958, **64** (7–8), 245–247.—Oxidations by means of ferricyanide (**I**) are accelerated when OsO_4 is used as a catalyst. The method makes possible the determination of hypophosphite and phosphite, and probably of formaldehyde, Cr^{III}, tellurite, sodium formaldehyde bisulphite and elementary S. Various sulphur compounds can also be determined and the theory of some of these determinations is discussed. For the simultaneous estimation of SO_3^{2-} (**II**), $S_4O_6^{2-}$ (**III**) and $S_2O_3^{2-}$ (**IV**), first oxidise **III** without catalyst with **I** to **II**; **II** and **IV** do not react. At higher pH (in 4 to 4·5 *N* alkali) in the presence of OsO_4 they are all oxidised to SO_4^{2-}. In a slightly acid iodine soln. **II** is oxidised to SO_4^{2-} and **IV** to **III**. The excess of iodine is destroyed by **II** and the **III** which has been formed is determined with **I** as already described. From the three titrations with **I**, the concn. of **II**, **III** and **IV** can be calculated. All end-points were determined by the dead-stop method. A. G. PETO

1614. **New titrimetric methods by precipitation.** E. Bakács (née Polgár) and L. Szekeres (Chem. Inst. of the Vet. Coll., Budapest). *Magyar Kém. Foly.*, 1958, **64** (7–8), 232–233.—Two types of indicator, were used in the pptn. methods—Eriochrome black T (**I**), *i.e.*, a metallochromic indicator and phenolphthalein (**II**). In aq. ethanol in the presence of **I**, PO_4^{3-} or AsO_4^{3-} can be determined with $MgSO_4$, Mg^{2+} with KH_2PO_4, and SO_4^{2-} with $BaCl_2$ at pH 10 (in a NH_4Cl - aq. NH_3 buffer) if a trace of Mg - EDTA is present. Alkali and other metal sulphates can be titrated with $BaCl_2$ in aq. ethanol in the presence of **II** and a known amount of Na_2CO_3 soln. In the simultaneous determination of OH^- and CO_3^{2-} in the presence of **II**, precipitate CO_3^{2-} with an excess of $BaCl_2$ (**III**) and titrate OH^- with $ZnCl_2$ soln. Three methods are described for the simultaneous determination of CO_3^{2-} and HCO_3^- even in a 0·1 *N* concn. If the normality of CO_3^{2-} is not more than half that of HCO_3^-, add NaCl or KCl, followed by an excess of **III**; titrate HCO_3^- with 0·1 *N* NaOH in the presence of **II**. If the concn. of CO_3^{2-} is greater than that of HCO_3^-, titrate CO_3^{2-} with **III** in aq. ethanol in the presence of **II**. Then add a known vol. of 0·1 *M* **III** in excess, boil the soln. for 2 to 3 min. and in a cooled 20 to 30% ethanolic soln. back-titrate the excess of **III** with 0·1 *N* Na_2CO_3. If the concn. of CO_3^{2-} is approx. equal to that of HCO_3^- (and not greater than twice the concn. of HCO_3^-), add an excess of a known NaOH soln. followed by an excess of **III**. Back-titrate the excess of NaOH with $ZnCl_2$. A. G. PETO

1615. **The use of Complexone IV (1:2-diaminocyclohexanetetra-acetic acid) in complexometry.** E. Kőrös and I. Poczók (Inst. Inorg. and Anal. Chem., L. Eötvös Univ., Budapest). *Magyar Kém. Foly.*, 1958, **64** (7–8), 250–252.—The Fe^{III} complexes of Complexone IV (**I**) are stable up to pH 13 at 20°, and are also stable towards triethanolamine at pH 10 to 12. For the analysis of a soln. containing Fe^{3+}, Ca^{2+} and Mg^{2+} (*e.g.*, as in dolomite), acidify it to pH 2 to 3 and titrate Fe^{3+} with **I** at 60° in the presence of sulphosalicylic acid. Cool the soln., make it alkaline and titrate the Ca^{2+} with EDTA (disodium salt) to murexide. Acidify the soln. with HCl to destroy the murexide, add aq. NH_3 and titrate the Mg^{2+} with EDTA (disodium salt) to Eriochrome black T. If Al is present, it can be masked with triethanolamine. Manganese can also be determined with **I** at pH 6 to 6·5 with hexamine (**II**) as buffer and methylthymol blue as indicator. In the same soln. Fe^{2+} can also be determined: Fe^{3+} are reduced with ascorbic acid and titrated at pH 5 to 6·5, in the presence of **II**, with **I** or EDTA (disodium salt). The alkaline-earth metals can be determined afterwards with **I** at pH 11. If other heavy metals are also present, titrate Fe in an aliquot at pH 2 to 3 with **I** or EDTA; reduce another aliquot with ascorbic acid and titrate in it the total of Fe and other heavy metals, and finally, at pH 11, the alkaline-earth metals. For the last two titrations, use **I** and methylthymol blue. A. G. PETO

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

1616. **Use of organic derivatives of selenous and tellurous acids in analysis.** I. P. Alimarin and V. S. Sotnikov (Anal. Chem. Dept., Moscow Univ.). *Vestn. Moskov. Univ.*, 1957, (6), 137–145.—The reactions of various aliphatic and aromatic derivatives of H_2SeO_3 and H_2TeO_3 with several cations are described. Possible uses are indicated. Thus Zr can be separated from all other elements except Sn, Ti, Nb and Ta by pptn. with ammonium benzene- or naphthalene-selenite from strongly acid soln., and if H_2O_2 is added only Sn interferes. G. S. SMITH

1617. **Spectrographic methods of determining traces of impurities. I. Chemical spectrographic methods of analysis of strontium, chromium and silicon. II. Spectrographic analysis of water and micro-samples by use of a strontium nitrate base.** Sh. I. Peizulaev, A. G. Karabash, L. S. Krauz, F. A. Kostareva, N. I. Smirnova-Averina, F. L. Babina, L. I. Kondrat'eva, E. F. Voronova and V. M. Meshkova. *Zavod. Lab.*, 1958, **24** (6), 723–731.—*Analysis of strontium*—The method depends on incomplete pptn. of Sr as $SrSO_4$, and concentration of the impurities in the smaller amount of $SrSO_4$ obtained by evaporation of the mother liquor. Standards based on $SrSO_4$ are prepared and the line intensities of 18 elements are determined in the d.c. arc spectrum. *Analysis of chromium*—Most of the Cr is volatilised as CrO_2Cl_2, the soln. is treated with $Sr(NO_3)_2$ and a residue based on $SrSO_4$ is obtained. From a d.c. arc spectrum 19 elements are determined. *Analysis of silicon*—The Si is volatilised as SiF_4 and 18 elements are determined in the d.c. arc spectrum after introduction of Sr. *Analysis of water*—The sample is evaporated in a platinum dish containing $Sr(NO_3)_2$, and the dried residue is excited in a d.c. arc to give a spectrum in which 12 elements are determined.
G. S. Smith

1618. **Use of amines in inorganic polarography. VII. Polarographic determination of copper and iron in nickel and aluminium and their salts.** J. Doležal and J. Novák (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (7), 1353–1354.—*Procedure*—Decompose the sample (0·5 g for ≃ 1% of Cu and 2% of Fe, 4 to 5 g for ≃ 0·05% of Cu and 0·1% of Fe) in HNO_3, transfer the soln. to a 100-ml flask and dilute to vol. Add 5 ml to each of two 25-ml flasks, adjust the pH to about 6 with NaOH soln., add 2 *M* ethylenediamine tartrate (10 ml) and *M* $K_4P_2O_7$ (2 ml) and adjust the pH again (the formed ppt. dissolves). To one flask add standard soln. of Cu^{2+} and Fe^{3+} and dilute the contents of both flasks to vol. Transfer to the polarographic vessel, remove O with N and register the wave from 0 to − 0·7 V. The method is suitable for the determination of ≮ 0·05% of Cu and 0·1% of Fe in nickel and ≮ 0·5% of Cu and 1% of Fe in aluminium.
J. Zýka

1619. **The simultaneous determination of arsenate, iodate, periodate and bromate ions.** L. Szekeres (Chem. Dept., Univ. for Agric. Sci., Budapest). *Magyar Kém. Foly.*, 1958, **64** (7–8), 230–232.—To determine AsO_4^{3-} (**I**), IO_3^- (**II**), IO_4^- (**III**) and BrO_3^- (**IV**) in the same soln., take 4 aliquots. To the first add 2% KI soln. and sufficient H_2SO_4 to make it 0·5 *N*. The iodine liberated by **II**, **III** and **IV** is titrated with $Na_2S_2O_3$ soln. To determine **III**, add $NaHCO_3$ and H_2O_2 to the second aliquot and warm it; this reduces **III** to **II** and decomposes the excess of H_2O_2. Acidify the soln to 0·5 to 0·8 *N* with H_2SO_4, add 2% KI soln. and titrate the iodine with $Na_2S_2O_3$ soln. From the difference between the first and second titrations, the concn. of **III** can be calculated. To the third aliquot, add solid KBr and H_2SO_4. This reduces **IV** to Br and **III** to **II**. Neutralise the soln. with NaOH and add $NaHCO_3$; reduce the BrO^- formed with H_2O_2 or urea. Any excess of the former is destroyed by heating; the latter does not interfere. Acidify with H_2SO_4, add 2% KI soln. and titrate with $Na_2S_2O_3$ soln. As the concn. of **III** is known, **II** can now be calculated. In the fourth aliquot reduce the halogenates to halides by adding solid KBr and 4% KI soln. to the acidified soln. Thus ultimately iodine is formed; add H_2O_2 and NaOH to convert iodine into I^-. Destroy the H_2O_2 by heating, strongly acidify the soln. with conc. HCl, add a few grams of solid KI and after 15 min. titrate with $Na_2S_2O_3$ soln. the iodine liberated by **I**. The error for the individual ions is < 1·5%. A mixture containing **II**, **III**, **IV** and CrO_4^{2-} (**V**) is analysed similarly, but in the first aliquot all four are determined. In the second one, determine **III** as before. From the third aliquot, by the same method as above, determine **V**, **III** and **IV** simultaneously by reducing **IV** to Br and **III** to **II** and Br by means of Br^-. The Br is determined as earlier; from this, calculate **III** + **V**. In the fourth aliquot, reduce all four ions with iodide in acid soln. Add H_2O_2 and make the soln. alkaline; this reduces iodine to iodide and oxidises Cr^{3+} to **V**; decompose the H_2O_2 and titrate **V** iodimetrically. If **V** is to be determined in the presence of **I**, add Br^- and H_2SO_4. From these, **V** liberates Br which can be directly titrated with standard As_2O_3 soln. in the presence of starch - iodine.
A. G. Peto

1620. **Analytical application of the determination of ferricyanide with ascorbic acid. I. Determination of ferrocyanide, chromate and permanganate.** L. Erdey and G. Svehla (Inst. f. allg. Chem., Tech. Univ., Budapest). *Z. anal. Chem.*, 1958, **163** (1), 6–12.—The titration of ferricyanide with ascorbic acid (**I**) (*Anal. Abstr.*, 1956, **3**, 3055) is used in this indirect procedure. Ferrocyanide is oxidised to ferricyanide with HNO_2 and the excess of HNO_2 is destroyed with sulphamic acid. Chromate and permanganate form equivalent amounts of ferricyanide by the oxidation of excess of ferrocyanide. The determination is completed in each case by titration with **I**, with 2:6-dichlorophenolindophenol as indicator. The accuracy of the ferrocyanide method is within 0·6% and the chromate and permanganate methods within 0·2% using 0·1 *N* **I**.
W. T. Carter

1621. **Spectrographic methods of determination of niobium, tantalum, zirconium, titanium, beryllium and indium.** N. A. Nomokonova and I. F. Morozova. *Trudy Vses. Magadansk. Nauch.-Issled. Inst., Min. Tsvetn. Metallurg. SSSR*, 1957, [4 (19)], 14–20; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,505.—For the determination of Nb, Ta, Zr and Ti, to 1 g of powdered carbon add 1 ml of a 2% soln. of ammonium molybdate, dry, and ignite at 400°. Mix the samples and standards with the prepared carbon powder in the ratio 1:1, the mixtures being introduced in craters drilled in carbon electrodes. Excite the spectra with an a.c. arc. Construct calibration curves on the co-ordinates ΔS and log *C*, samples of the ores or artificial mixtures being used. Niobium and Ta are determined together; the determinable concn. are 0·03 to 2·0% for Nb and Ta and 0·12 to 2·0% for Zr. The analytical lines used are Nb 2950·9 and Mo 2930·5, Ta 2685·1 and Mo 2930·5, Zr 2571·4 and Mo 2683·2, and Ti 3078·6 and Mo 2930·5 A. The error of the analysis is ± 8%. To determine Be and In, introduce 100 to 200 mg of the sample on paper strips into the flame of the arc with horizontal electrodes. To the samples or standards (2 g) add 1 ml of a 1·5% soln. of Cr as internal standard for Be; as internal standard for In use Bi (2%) or Cd (5%). Dip the strips of paper, length 20 cm, in $(NH_4)_2SO_4$ soln. and introduce into the flame for 100 to 120 sec. with current 8 amp. The analytical lines are Be 2348·6 and Cr 2855·7 (for

concn. 0·001 to 0·2%); Be 2650·5 and Cr 2677·2 (for concn. 0·2 to 2·0%); In 2710·3, 3039·3, 3256·1 and 3258·5; Bi 2898·0; Cd 3261·1; and Sb 2877·9 A. The error of the analysis is ± 8%. The presence of Ge and Mn may introduce line interference. The position of the calibration curves depends greatly on the over-all composition of the samples.

C. D. KOPKIN

1622. **Method of repeated radioactive dilution for determining small amounts of impurities.** I. E. Zimakov and G. S. Rozhavskii (State Sci. Res. Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1958, **24** (8), 922–925.—The method avoids the need for determining specific activity and permits the determination of impurities in amounts of $\simeq 10^{-7}$%. Its application to the determination of $\simeq 10^{-5}$% of Sb in lead is described. If two equal aliquots, each containing x mg of the element to be determined, are treated with m_1 and m_2 mg, respectively, of a radioactive isotope of the element with specific activity a_0, which need not be known, and exactly equal amounts of the element are extracted from each and their activities measured, then, since the activity in each case is $A_i = a_0 m_i y/(m_i + x)$, where y is the extracted amount and i is 1 or 2, the value of x can be found from the equation $x = m_1 m_2 \times (A_2 - A_1)/(A_1 m_2 - A_2 m_1)$. For greatest accuracy m_1 is approx. equal to x, and m_2 is much greater than x, or conversely. With m_1 not equal to x, the accuracy decreases as the value $|(m_1 - x)|/(m_1 + x)$ increases. By the use of several aliquots and various amounts of m_i, results of high accuracy can be obtained. Thus if x is assumed to lie between m_1 and $100m_1$, one can use five aliquots and introduce m_1, $4m_1$, $12m_1$, $36m_1$, and $108m_1$, and there will always be obtainable the conditions $|(m_i - x)|/(x + m_i) > \frac{1}{2}$ and m_i much greater than x. For determining minute traces of Sb in lead, the sample soln. obtained by dissolution in a mixture of HNO_3 and tartaric acid is divided into n equal parts and various amounts (m_1 to m_n) of radioactive Sb dissolved in a similar acid mixture are added. All the soln. are treated with HCl so that the acidity corresponds to a 3:1 mixture. After reduction with $SnCl_2$, followed by addition of $NaNO_2$ and then urea, the soln. are diluted to an acidity of 1:9 in HCl and treated with methyl violet soln. in amount sufficient for combining with only part of the Sb. Immediate extraction with toluene gives soln. all containing the same amount of Sb. The activities are measured and the content of Sb in the sample is calculated from the equation given.

G. S. SMITH

1623. **Determination of hydrogen in tantalum, niobium and uranium.** A. N. Zaĭdel' and K. I. Petrov. *Zavod. Lab.*, 1958, **24** (8), 1000–1001.—The method previously described (*cf. Anal. Abstr.*, 1957, **4**, 865) is applied to tantalum, niobium and uranium. The method is sensitive to 10^{-4}% by wt. on a sample wt. of 1 g.

G. S. SMITH

1624. **Rapid determination of water vapour in gases by means of detector tubes.** Yoshitaka Kobayashi (Fac. of Engng, Yokohama Univ., Minami-ku). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (6), 679–682.—Four detector tubes [silica gel containing Se and H_2SO_4 (Grosskopf, *Angew. Chem.*, 1951, **63**, 306), alumina containing $CoCl_2$, and silica gel containing crystal violet, $MnSO_4$ and $ZnCl_2$ (Demidov, *Gigiena i Sanit.*, 1957, **2**, 64), or malachite green, $MnSO_4$ and $ZnCl_2$ (Demidov, *loc. cit.*)] were examined with reference to the interference of other gases. The first is suitable for the analysis of a micro amount of H_2O, but interference results from acetylene, acetone, ethanol, ether, acetic acid and its esters, NH_3, HCN, H_2S and HNO_3. The second is little affected by other gases except H_2S. The last two are rather sensitive towards org. vapours except acetylene, CCl_4 and trichloroethylene. The estimation can be effected either by the measurement of the length of the coloured layer or by that of the sample vol. required for the development of a given length of the coloured layer.

K. SAITO

1625. **Routine assay of tritium in water and labelled substances in the range 20 to 10^4 $\mu\mu$C.** P. R. Payne and J. Done (Nat. Inst. for Med. Res., London). *Phys. in Med. Biol.*, 1958, **3**, 16–26.—A method is presented for the determination of radioactive hydrogen in a proportional gas counter, and conditions for the best operation of the counter are described. The apparatus, which has been in use for 8 years, is easy to operate and requires very little maintenance. The gas filling for the counter is produced by the reaction of active water on aluminium carbide in a simple metal vessel, and this is extended to the assay of tritium in labelled compounds, by a procedure consisting of a preliminary combustion in a high-pressure oxygen bomb, followed by the reaction of the water produced with aluminium carbide. The whole procedure takes less than 1 hr., and any required number of samples can be prepared at the same time. The method is accurate to at least ± 1% over the range of activities 20 to 10^4 $\mu\mu$C.

NUCL. SCI. ABSTR.

1626. **The photometric and conductimetric titration of lithium in *cyclo*hexanone or acetone.** H. Specker, H. Hartkamp and E. Jackwerth (Inst. for Spectrochem., Dortmund). *Z. anal. Chem.*, 1958, **163** (2), 111–119.—Solutions of lithium chloride in acetone, *cyclo*hexanone or other carbonyl-containing organic solvents form a red-orange complex when treated with a solution of a cuprous salt in acetone, and this reaction has been applied to the determination of lithium. The alkali chloride sample after drying at 120° to 130° is extracted for 1 hr. with 50 ml of acetone or *cyclo*hexanone, or refluxed for 20 min. with the same amount of solvent. The extract is filtered and the filtrate made up to 100 ml, from which 5-, 10- or 25-ml portions are taken and placed in a titration cell. Titration is carried out with a standard 0·01 or 0·001 *M* solution of copper perchlorate in acetone, the end-point being determined either photometrically or conductimetrically. Typical curves are shown for various concentrations of lithium chloride. The method is applicable in the range 10^{-3} to 10^{-6} moles of Li, and by using microchemical methods may be extended down to 10^{-6} to 10^{-9} moles of Li. The presence of NaCl and KCl does not lead to errors since they are removed in the preliminary extraction stage. Addition of benzene sharpens the end-point for very small quantities of lithium.

S. M. MARSH

1627. **Determination of traces of oxygen in sodium metal by infra-red spectrophotometry.** H. J. de Bruin and L. E. Smythe (Atomic Energy Commission, Sutherland, New South Wales, Australia). *Nature*, 1958, **182**, 387.—From 5 to 100 p.p.m. of Na_2O in sodium metal can be determined by treating the sample (2 g) with alkyl bromide (*cf.*

Anal. Abstr., 1954, **1**, 1244), dissolving the reaction mixture in H_2O, and converting the unchanged oxide (now NaOH) into $NaHCO_3$ by passing in an excess of CO_2. The soln. is then freeze-dried and the powder is heated at 400° for 2 hr. A compressed disc is made from 1500 mg of the $NaBr - Na_2CO_3$ mixture, and the absorbance of the Na_2CO_3 is measured at 11·38 μ in a Perkin-Elmer model 21 infra-red spectrometer. Although Beer's law is not strictly valid, the concn. of oxygen in the sample can be calculated from a standard curve to within $\simeq$ 10 p.p.m. by wt.

W. J. BAKER

1628. **Gravimetric determination of potassium with hexanitrodiphenylamine (dipicrylamine).** S. H. Iqbal and G. D. Bhat (Central Salt Res. Inst., Bhavnagar). *J. Sci. Ind. Res., India, B*, 1958, **17** (8), 307–309.—A simple and reliable method for the semi-micro determination of K as dipicrylaminate is described. The limitations of the method and the effect of cations such as Na^+, Mg^{2+} and Ca^{2+}, and of anions such as SO_4^{2-} and NO_2^- on its accuracy are described. I. JONES

1629. **Indirect potentiometric titration of potassium, rubidium and caesium with the sodium salt of tetraphenylboron.** J. Havíř (Milit. Tech. Acad., Brno, Czechoslovakia). *Chem. Listy*, 1958, **52** (7), 1274–1278.—In this determination, the ppt. formed from the sodium salt of tetraphenylboron (**I**) and K^+, Rb^+ or Cs^+ is dissolved in acetone, and **I** is determined potentiometrically with the use of $AgNO_3$ soln. The influence of acetone, acidity and of electrolyte as well as of Cl^-, Br^- and I^- has been studied. *Procedure*—To the soln. of the sample containing $\gtrsim$ 10 mg of K, Rb or Cs (in the form of chloride or sulphate) in 100 ml add **I** soln. (2%). Collect the ppt. in a crucible (A1), and wash it with a satd. soln. of the potassium, rubidium or caesium salt of tetraphenylboron and with a small vol. of H_2O. Transfer the main portion of the ppt. to the vessel in which the procedure has been carried out, dissolve the remaining portion in water-free acetone, combine with the ppt. and add acetone (20 to 30 ml) till dissolution is complete. Dilute with the same vol. of H_2O and titrate with 0·1 *N* $AgNO_3$ (freshly prepared silver indicator electrode, satd. calomel reference electrode immersed in 10% $NaNO_3$ soln.; the salt bridge is filled with a 2% soln. of agar in 10% $NaNO_3$ soln). The titration must be carried out dropwise; when a potential of 150 mV is reached, the volumetric reagent must be added in equal known volumes. The potential change depends on the concn. of acetone. Dilute mineral acids do not interfere. Alkali halides must be absent when titrating the separated ppt. The value of the solubility product of the Ag tetraphenylboron ($4·0 \times 10^{-14}$) has been found experimentally. J. ZÝKA

1630. **Capture of radioactive caesium on to molybdophosphate precipitate and its determination.** Tadashi Hara (Doshisha Univ., Kyoto, Japan). *Bull. Chem. Soc. Japan*, 1958, **31** (5), 635–640 (in English).—Up to 500 μg of ^{137}Cs in $\gtrsim$ 5 litres of soln. can be quant. captured on $\simeq$ 160 mg of pptd. thallium molybdophosphate, the activity of the ppt. then being proportional to the contained ^{137}Cs. The accuracy is higher than for the usual co-pptn. method. Only Rb ($>$ 8 mg per 100 ml), Tl, WO_3^{2-} and NO_2^- interfere seriously by co-capture and reduction of the counts; interference by ^{90}Sr and rare-earth elements can be avoided. *Procedure*—Molybdophosphoric acid (20 ml) is added to a mixture of $TlNO_3$ soln. ($\equiv$ 1 mg of Tl per 1 ml of 2% HNO_3) (50 ml), conc. HNO_3 (4 ml) and H_2O (126 ml), and the soln. is concentrated to $\simeq$ 7 ml. The ppt. is collected on a glass filter disc, washed with 2% HNO_3 and the sample soln. (in 2% HNO_3) is passed through the ppt. at $\simeq$ 2 drops every 3 sec. The ppt. layer is then washed with 2% HNO_3, followed by conc. acetic acid, and its activity is measured after drying for $\simeq$ 1 hr. at 120°.

W. J. BAKER

1631. **Colorimetric determination of copper(II) using sodium alizarin-3-sulphonate.** Anil K. Mukherji and Arun K. Dey (State Univ., Baton Rouge, La., U.S.A.). *Bull. Chem. Soc. Japan*, 1958, **31** (5), 521–524 (in English).—Trace amounts of Cu^{2+} can be determined spectrophotometrically by measuring the extinction at 500 mμ of the 1:1 complex with 0·001 *M* Na alizarin-3-sulphonate (alizarin red S) formed at pH 4 to 6 and 20° to 30°. A tenfold excess of the reagent is necessary, and the lower limit of detection is 6·4 p.p.m. of Cu in a 1-cm cell. Interfering ions are listed, with tolerance limits. W. J. BAKER

1632. **Photometric determination of copper with diacetyldi(thiobenzoylhydrazone).** G. Bähr and D. Thiele (Inst. f. Anorg. Chem., Ernst-Moritz-Arndt Univ., Greifswald). *Chem. Tech., Berlin*, 1958, **10** (7), 420.—In 50% H_2SO_4, diacetyldi(thiobenzoylhydrazone) forms a stable complex with Cu, extractable with $CHCl_3$, having absorption maxima at 530 mμ and 375 mμ, and a linear Cu concn. *vs.* absorption curve in the range 6 to 488 μg per ml. Arsenic, Sb and Au also form $CHCl_3$-soluble complexes, but do not interfere if the ratios of As:Cu, Sb:Cu, and Au:Cu are less than 40, 4 and 1, respectively. J. L. PROSSER

1633. **Water-immiscible organic solvents in flame photometry. Determination of copper by extraction as a complex.** H. Bode and H. Fabian (Anorg.-chem. Inst. Tech. Hochschule, Hanover, Germany). *Z. anal. Chem.*, 1958, **162** (5), 328–336.—The effect of the solvent on the sensitivity of the flame-photometric determination of Cu is reported for 40 different organic solvents in which were dissolved such complexes as Cu diethyldithiocarbamate, cupferronate and oxinate. Aliphatic esters and ketones, methyl phenyl ether, benzene, toluene and xylene are effective solvents. With the procedure described and any one of the four last-named solvents, the sensitivity can be increased $\simeq$ 40-fold in comparison with the usual method in which the aq. soln. containing Cu is sprayed into the flame. The initial extraction of the Cu complex by the solvent is rapid and complete, especially with benzene, toluene or xylene. The optimum ratio of the pressure of O to that of H is a critical factor; preferred values are listed. The spray time is $\simeq$ 100 sec. for 5 ml of solvent. W. J. BAKER

1634. **Dialkyl and diaryl phosphorodithioates as analytical reagents. V. Photometric determination of copper.** A. I. Busev and M. I. Ivanyutin (Anal. Chem. Dept., Moscow Univ.). *Vestn. Moskov. Univ.*, 1957, (5), 157–161.—The soln. ($\simeq$ 50 ml) acidified with 1 to 2 ml of 2 *N* H_2SO_4 is shaken with 5 to 10 ml of CCl_4 and a 2 to 8-fold excess of 0·001 *M* K or Ni diethyl phosphorodithioate, the extract is removed and the aq. layer is further extracted with CCl_4 (3 $\times$ 5 ml). The extinction of the combined extracts after filtration and dilution to 25 ml is measured at 420 mμ to

determine Cu. The complex has the formula $[(C_2H_5O)_2PSS]_2Cu$. There is no interference from Pb, Zn, Cd and Al. To determine Cu in aluminium (0·005% of Cu), cadmium (0·007% of Cu), zinc (0·001% of Cu), nickel (0·013% of Cu), and steel (0·2 to 0·3% of Cu), the sample is dissolved in HNO_3, followed by boiling with H_2SO_4, or in H_2SO_4, and an aliquot of the diluted soln. is diluted so that the concn. of Cu is $\geq$ 2 mg per litre. The reagent and solvent are added and the extraction, etc., is carried out as described above.

G. S. SMITH

1635. **New volumetric method for the determination of copper with potassium ferrocyanide.** A. S. Kozlov and V. V. Bagreev. *Nauch. Dokl. Výssh. Shkolÿ Khim. i Khim. Tekhnol.*, 1958, (2), 300–304; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,510.—The method is based on the pptn. of Cu^{2+} with $K_4Fe(CN)_6$ (**I**) as $(NH_4)_2CuFe(CN)_6$ from a weakly ammoniacal soln. in the presence of ammonium salts. After removal of the ppt., the excess of **I** is titrated with $ZnSO_4$, with diphenylamine as indicator. A comparison of the results of determining Cu in malachite, standard samples of oxidised copper ore and tails of copper flotation by the proposed and the iodimetric methods indicates no marked difference (the discrepancy for malachites is $\geq$ 0·24%, and in the other cases even smaller). Metals that form insol. ferrocyanides with **I** interfere.

C. D. KOPKIN

1636. **2-Methylbenziminazole as analytical reagent for silver.** Prabhab K. Misra and Birendra K. Patnaik (Mayurbhanj Chem. Lab., Ravenshaw Coll., Cuttack-3). *J. Indian Chem. Soc.*, 1958, **35** (7), 519–520.—2-Methylbenziminazole is used as a reagent for the gravimetric determination of Ag. The complex has the formula $Ag(C_8H_7N_2)$. The addition of ammoniacal EDTA soln. prevents the co-pptn. of Cu^{2+}, Ni^{2+}, Mg^{2+} and Co^{2+}.

I. JONES

1637. **Determination of silver in alloys by radiometric titration.** V. I. Plotnikov (All-Union Mineral-Metallurgical Sci. Res. Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1958, **24** (8), 927–928.—The sample soln. (100 ml) containing added ^{110}Ag and having an initial activity of $\geq$ 2500 counts per min. per 25 ml is titrated with 0·005 or 0·1 *N* HCl with periodical measurement of the activity of a 25-ml aliquot temporarily sucked up into a vessel for the purpose. The end-point is determined graphically. With alloys, copper- and lead-based, the sample (20 g) is dissolved in conc. HNO_3 and a suitable aliquot of the filtered soln. is treated with radioactive Ag, the acidity is made $\simeq$ 10% in HNO_3, and the titrant is added.

G. S. SMITH

1638. **Ion-exchange separation of beryllium by elution with salicylate analogues.** J. Schubert, A. Lindenbaum and W. Westfall (Argonne Nat. Lab., Lemont, Ill.). *J. Phys. Chem.*, 1958, **62** (4), 390–394.—Trace quantities of Be are separated quant. from macro-concn. of salts by means of a cation-exchange resin. Beryllium is eluted selectively with sulphosalicylic acid (**I**) (0·02 to 0·10 *M*) at a pH of 3·5 to 4·5. Neither Cu^{2+}, UO_2^{2+} nor Ca^{2+} are removed under these conditions. Uranyl ion is eluted by **I** at a pH of 4·5 to 4·7. Foreign cations are subsequently removed by elution with HCl or H_2SO_4. At pH > 6 and in the presence of **I**, Be is strongly adsorbed by an anion-exchange resin. It is shown that Be forms an uncharged complex with **I** in the acid range and a negatively charged complex in the neutral and alkaline ranges.

K. R. C.

1639. **Complexometric determination of magnesium in aluminium alloys.** K. Študlar and I. Janoušek (Res. and Control Inst. ZVIL, Plzeň, Czechoslovakia). *Hutn. Listy*, 1958, **13** (7), 643–646.—*Procedure*—Dissolve the sample (0·2 to 5 g equiv. to 6 to 0·1% of Mg) in NaOH soln. (30%) (10 to 50 ml), dilute with hot water to 200 ml, add moistened filter-paper, mix and set aside. Warm and filter through filter-paper and wash the ppt. three times with hot water. Wash the ppt. from the filter and dissolve any residue on the filter with hot H_2SO_4 (5%) (15 ml). Warm for 2 to 3 min., add NH_4Cl soln. (25%) (25 ml) and dilute with hot water to 150 ml. Add saturated bromine water (25 ml) (oxidation of Mn) and mix, then add aq. NH_3 (1:1) carefully till the orange soln. is decolorised. Add aq. NH_3 in excess (1 to 2 ml), warm, filter through a filter-paper and wash 4 or 5 times with hot water. Add conc. aq. NH_3 (15 ml), a small amount of KCN, and Eriochrome black T as indicator, and titrate with 0·05 *M* EDTA (disodium salt) till blue.

J. ZÝKA

1640. **Elimination of anionic interference in flame-photometric determination of calcium in the presence of phosphate and sulphate.** J. Yofè and R. Finkelstein (Atomic Energy Commission, Tel Aviv, Israel). *Anal. Chim. Acta*, 1958, **19** (2), 166–173.—The emission of Ca^{2+} at 422·7, 554 and 662 mμ is depressed by increasing addition of PO_4^{3-} until the PO_4^{3-}:Ca^{2+} mol. ratio reaches 1, when a const. emission is obtained, unaffected by further addition of PO_4^{3-}. The original emission of Ca^{2+} at 422·7 mμ is restored when La^{3+} are added in the mol. ratio La^{3+}:PO_4^{3-} = 5·1:1, or in the ratio 3·5:1 for λ = 554 mμ or 4·1:1 for λ = 622 mμ. The recovery is discussed in terms of the probable course of the flame reactions and is attributed to the fact that, of Ca and La, La forms the more thermostable phosphate. Interference by SO_4^{2-} reaches a max. for λ = 554 mμ when SO_4^{2-}:Ca^{2+} = 1·5:1 and the addition of La^{3+} in the ratio La^{3+}:SO_4^{2-} = 6 to 7:1 restores the emission. The addition of Fe^{3+} also counters the depressive effect of PO_4^{3-}, but a high proportion, 16:1 for λ = 554 mμ, is required.

H. N. S.

1641. **Analytical use of phenolcarboxylic acids of the triphenylmethane series. Rapid determination of calcium and magnesium in rocks.** I. S. Mustafin and E. A. Kashkovskaya. *Nauch. Dokl. Výssh. Shkolÿ Khim. i Khim. Tekhnol.*, 1958, (2), 297–299; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,523.—The dyestuff Chromoxan green GG (**I**), recommended earlier as indicator for the complexometric titration with EDTA of total Ca and Mg (*Anal. Abstr.*, 1957, **4**, 3524), is used for the rapid determination of Ca and Mg in rocks and minerals. After removal of the oxides of tervalent metals, the total Ca and Mg is titrated, with **I** as indicator; the content of Ca is determined by titration in the presence of murexide.

C. D. KOPKIN

1642. **Analytical applications of sensitivity diagrams. III. Semi-quantitative determination of calcium in potassium dichromate.** F. Burriel-Martí, J. Ramírez-Muñoz and M. L. Rexach-M. de Lizarduy (Univ. C.S.I.C., Madrid). *Inf. Quim.*

Anal., 1958, **12** (3), 61–67.—The content of Ca in samples of potassium dichromate can be found by preparing a 10% (w/v) soln. and diluting until the turbidity due to $CaCr_2O_7$ just disappears. The dilution is measured and the concn. of Ca is found from a standard curve. The method is applicable to samples containing 0·01 to 0·5% of Ca. Results are 75 to 95% of theoretical. G. H. FOXLEY

1643. **The effect of phosphorus on the emission of calcium in a flame.** C. T. J. Alkemade and M. H. Voorhuis (Rijks-Univ., Utrecht). *Z. anal. Chem.*, 1958, **163** (2), 91–103.—After a review of previous work on the depression of calcium emission in flame photometry by the presence of phosphorus, a systematic investigation of the phenomenon is described. Results are presented which indicate the effects of added organic compound, height of measurement in the flame, temperature of the flame and calcium concn. on the emission. No depressive effect was observed when pure soln. of Ca and of P were injected into the flame simultaneously from parallel atomisers. A mechanism is proposed to explain the depressive effect. The depressive effect of phosphorus can be reduced to negligible proportions by (*a*) greatly diluting the P-containing soln. of Ca, (*b*) measuring at a high point in the flame (where dissociation nears completion), (*c*) using a high flame temperature, (*d*) using an atomiser which gives fine dispersion. If the depressive effect is to be used as an indirect method for the determination of P, care should be taken to see that the effect is independent of calcium concn. and varies with the height in the flame. S. M. MARSH

1644. **Paper-chromatographic determination of barium and strontium in the evaporation products from oxide cathodes.** W. Düsing and H. Kunze (Telefunken G.m.b.H., Ulm, Germany). *Z. anal. Chem.*, 1958, **162** (5), 345–356.—The sample is dissolved in a mixture (2 + 1) of 4 *N* HCl and methanol, the soln. is evaporated to dryness and the residue is dissolved in a few ml of dil. HCl. By ascending paper chromatography of this soln. on strips developed with methanol - HCl - H_2O (8:1:1), separation of Sr from Ba and from other metals can be achieved. The spots are revealed as rhodizonates, and the concn. of Ba and Sr (0 to 30 μg of each) are determined photometrically at 510 mμ (Sr) and 525 mμ (Ba). Lead and Sr give similarly coloured rhodizonates, the R_F values being approx. equivalent. W. J. BAKER

1645. **Separation of radium from radium-D, radium-E and polonium with EDTA.** V. Vebersík and K. Horová (Radiolog. Res. Inst., Prague, Czechoslovakia). *Z. anal. Chem.*, 1958, **162** (6), 401–407.—The use of EDTA (disodium salt) permits the separation of Ra from its decay products Ra-D, Ra-E and Po. In weakly acid soln. (pH 4·5 to 5), only $RaSO_4$ is pptd. by the addition of $(NH_4)_2SO_4$. A further separation is carried out by dissolving the ppt. in an ammoniacal soln. of EDTA (disodium salt) followed by re-pptn. on acidification with acetic acid. Determination of the Ra by measurement of its γ-radiation after 6 weeks shows a loss of $\simeq 10^{-6}$ g of Ra per 100 g of H_2O, while β-radiation measurements on the separated Ra-D, Ra-E and Po show losses of $\simeq \pm$ 3%. J. H. WATON

1646. **Volumetric determination of zinc with potassium ferrocyanide, with the use of reversible redox-adsorption indicators.** J. Bognár and L. Nagy (Inst. Chem. II, Tech. Univ. of the Heavy Industries, Miskolc, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1958, **16** (1), 1–8.—This is an English version of an article published originally in Hungarian. (*Cf. Magyar Kém. Foly.*, 1956, **62** (7), 217; *Anal. Abstr.*, 1957, **4**, 405.) T. R. ANDREW

1647. **Spectrochemical determination of impurities in high-purity metallic zinc.** R. I. Gutkina (Sverdlovsk Filial of D. I. Mendeleev All-Union Sci. Res. Inst. of Metrology). *Zavod. Lab.*, 1958, **24** (6), 734–736.—The impurities are separated as sulphides from a soln. obtained by dissolving the sample in dil. HNO_3 and adding excess of aq. NH_3. The ppt. containing Pb, Fe, Cd and Cu is ashed, the residue is dissolved in HNO_3, and the soln. on a carbon electrode is excited in an arc. G. S. SMITH

1648. **The detection of cadmium as hexamethonium cadmium iodide.** M. Ziegler (Inst. f. anorg. Chem., Univ. Göttingen, Germany). *Z. anal. Chem.*, 1958, **163** (1), 27–29.—Cadmium forms a white insoluble complex $[(CH_3)_3N{\cdot}(CH_2)_6{\cdot}N(CH_3)_3][CdI_4]$ with hexamethonium chloride (**I**) in the presence of excess of alkali-metal iodide. The reaction is exceedingly sensitive and can be used for the detection of Cd in the presence of a 2000-fold excess of Zn, Ni, Co and Mn, or a 400-fold excess of Al. The limit of detection is 5 μg of Cd and the dilution limit is 1 in 3×10^5. *Procedure*—To the test soln., which should be neutral or buffered with Na acetate, add solid KI and a 10% aq. soln. of **I**. The formation of a white turbidity or ppt. indicates the presence of Cd. Oxidising agents which give a brown colour due to the formation of free iodine may be eliminated with SO_2 or $NaHSO_3$ before the addition of **I**. W. T. CARTER

1649. **Analysis of pure metals. Determination of traces of cadmium in indium and thallium.** V. A. Nazarenko and G. V. Flyantikova (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1958, **24** (7), 801–802.—To determine micro amounts of Cd in indium, the sample (0·5 g) is dissolved in a mixture of 5 ml of water, 0·5 ml of conc. H_2SO_4 and 2 ml of conc. HNO_3, and the cooled soln. is treated first with 15 ml of water and 3 g of tartaric acid, then with 1·5 ml of 25% KSCN soln. and 2 drops of 0·1% bromocresol purple soln., followed by dropwise addition of 20% NaOH soln. to give a bluish-violet colour and of dil. H_2SO_4 soln. (1:4) to give a yellow colour, and then with 10 ml of an acetate buffer soln. (pH 5). The soln. is extracted with pyridine - $CHCl_3$ (1:20) (3×5 ml), the extract is washed once with 15 ml of 2% KSCN soln., and evaporated carefully to dryness, and the residue after addition of 1 ml of conc. HNO_3, 0·1 ml of conc. H_2SO_4 and 0·2 ml of 2% $K_2S_2O_7$ soln. is again evaporated to dryness and then ignited at 400° to 450°. The cooled melt is dissolved in 0·02 *N* HCl ($2 \times 2{\cdot}5$ ml) and the soln. in a stoppered colorimeter tube is treated with 0·3 ml of 10% hydroxyammonium chloride soln., 2 ml of 10% NaOH soln. and 0·1 ml of 1% KCN soln., and then shaken for 0·5 min. with 1 ml of a 0·002% soln. of dithizone in $CHCl_3$. The colour is compared with that of a series of standards containing from 0 to 0·2 μg (0·05-μg intervals) and then to 1 μg (0·1-μg intervals). With thallium, the sample (0·5 g) is dissolved in 2 ml of conc. HNO_3 and the cooled soln. after dilution to 35 ml is poured slowly into 20 ml of 25% KSCN soln. The ppt. of TlSCN is filtered off, the filtrate is neutralised to

bromocresol purple indicator with 20% NaOH soln. and dil. H_2SO_4 (1:4), 10 ml of an acetate buffer (pH 5) is added, and the Cd is extracted as described above. Quantitative separation of 0·05 μg of Cd from 0·5 g of Tl is attainable. G. S. SMITH

1650. **A new method for the photometric determination of micro quantities of mercury(II) ions in aqueous solutions.** V. Karas and T. Pinter (Dept. Chem., Med. Fac., Univ. Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (2), 141–147 (in German).—In weakly acid soln. under the influence of light or of minute amounts of Hg^{II} the ferrocyanide ion decomposes to form Fe^{3+} and ultimately Prussian blue. In the presence of 1:10-phenanthroline (**I**) or 2:2′-dipyridyl (**II**), however, the red soluble complex $[Fe(\mathbf{I})_3]^{2+}$ or $[Fe(\mathbf{II})_3]^{2+}$ is formed, and the intensity of the red colour is proportional to the amount of Hg^{2+} present. This is suggested as the basis of a new method for the determination of Hg^{II}. Experiments have shown that for **I** the optimum pH is 3, and the max. absorption is at 510 mμ. If a soln. made up to 15 ml, heated to 60° for 5 min. before measurement of the colour, and containing HCl (2 ml of 0·01 *M*), **I** (2 ml of 0·006 *M*), $K_4Fe(CN)_6$ (1·5 ml of 0·001 *M*) and $HgCl_2$ (2 to 26 ml of 0·00001 *M*) is used it was shown that the Beer - Lambert law was obeyed. H. M.

1651. **The determination of mercury oxycyanide.** F. P. Wilson, P. H. B. Ingle and C. G. Butler (Dept. of Pharmacy, Bradford Inst. of Technol., England). *J. Pharm. Pharmacol.*, 1958, **10** (7), 442–446.—Dissolve the sample of mercuric oxycyanide (0·5 g) in H_2O (50 ml) and boil gently for 10 min. to expel CO_2. Cool to 20° without agitation, add KSCN (1 g) and determine HgO by titration with 0·1 *N* HCl to the full yellow colour of phenol red. To determine $Hg(CN)_2$, continue the titration after the addition of KI (4 g) and formaldehyde soln. (2 ml) from which formate ions have been removed by treatment with an anion-exchange resin (OH form). The coeff. of variation, based on 6 assays by each of 4 operators, is ± 1·17% for HgO and ± 0·23% for $Hg(CN)_2$. The mean results agree closely with those by the method of the B.P. 1953. A. R. ROGERS

1652. **Colorimetric determination of micro quantities of boron using curcumin.** A. D. Gol'tman and V. G. Gurevich. *Ukr. Khim. Zhur.*, 1958, **24** (2), 244–250; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,564.—The curcumin method for the determination of B is studied and improved to give increased accuracy. Evaporate the oxidised soln., containing H_3BO_3 and curcumin (**I**), at 50° to 60° in 50-ml crucibles, the soln. being kept alkaline with $Ca(OH)_2$. To the dry residue add a mixture of oxalic acid and HCl (0·363 *M* in oxalic acid and 1·3 to 2·4 *M* in HCl) and an ethanolic soln. of **I** (1 ml of soln. containing 100 μg of **I** for 0·8 μg of B); add **I** in fourfold molar excess based on H_3BO_3. In the photometric determination, the determinable concn. of B is 0·14 to 1·3 μg in 5 ml of soln.; the error is ≯ 10%. Interference is caused by the presence of oxalate, NO_3^-, SO_4^{2-}, Cl^-, HPO_4^{2-}, Ca^{2+}, Fe^{3+}, K^+, Mg^{2+} and Na^+ (particularly by NO_3^-, HPO_4^{2-}, K^+ and Fe^{3+}), which lower the intensity of colour. The mol. extinction coeff. with 2×10^{-5} *M* H_3BO_3 is $6{\cdot}3 \times 10^4$. The coloured compound contains B and **I** in the ratio 1:1. Using colorimetry by the standard series method, the error of the determination is ≯ 10%, with a sensitivity of 1 μg of B per litre. A procedure is given for the determination of B in vegetable material. C. D. KOPKIN

1653. **Emission spectrographic determination of metallic impurities in powdered boron.** S. Lader (Picatinny Arsenal, Dover, N.J., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (3), 85–86.—In concn. of up to about 1·3%, Al, Fe, Si, Cu, Sn, Cr, Ti, Mn, Pb, Ca and Mg can be determined with an average standard deviation of 10%. Samples from different sources gave a coeff. of variation for the impurities varying from 6% to 15%. A.c. arc excitation is used, with nickel as an internal standard. K. A. PROCTOR

1654. **Problems in trace analysis [of aluminium].** P. Urech (Forschungsinst. der Aluminium-Industrie, A.-G., Neuhausen). *Mitt. Lebensmitt. Hyg., Bern*, 1958, **49** (6), 442–447.—A critical review is made of methods available for the determination of trace elements with particular reference to Al, *viz*, colorimetry, emission spectrometry, flame photometry, polarography, activation analysis and mass spectrometry. Results obtained by colorimetry and activation analysis are compared for (*a*) determination of Cu, and (*b*) determination of Si, Fe, Cu, Zn, Mg, As, Mn, Ga, Ni, Na, N and C; and those obtained by colorimetry and mass spectrometry are compared for the determination of Si, Fe and Cu. Results obtained by mass spectrometry for the determination of 18 trace elements in three grades of aluminium are given and the sensitivities of the various analytical methods are tabulated for 25 trace elements. S. M. MARSH

1655. **Polarographic determination of aluminium. III. Preliminary study of the mechanism of the aluminium - Solochrome violet RS reaction.** M. Perkins and G. F. Reynolds (Min. of Supply, Chemical Inspectorate, Woolwich, England). *Anal. Chim. Acta*, 1958, **19** (1), 54–64.—The reactions were studied in the pH range 1·95 to 10·08 and two different complex species were noted, which were stable in the pH ranges 4·2 to 5 and 5·7 to 7·0. At pH 6·5 the molar ratio of Al to dye was 1:3, and, at pH 4·6, 1:2. Data on numbers of electrons per molecule transferred during the reduction of the complexes are presented.

IV. A study of the reduction of some hydroxyazo dyes in relation to the aluminium - Solochrome violet RS complex. M. Perkins and G. F. Reynolds. *Ibid.*, 1958, **19** (2), 194–201.—Following the trend of previous work (*cf.* Part III) with Solochrome violet RS, the polarographic reduction of three azo dyes, Orange II, Resorcin yellow and Acid Alizarin black SN, is investigated both with and without the addition of Al^{3+}. The results are examined in relation to the probable configuration of the complexes of Al with *ortho* mono- and di-hydroxyazo dyes. It is concluded that Al forms a true Werner complex with an *o*-hydroxyazo dye by replacement of the hydroxy group or groups and the acceptance of an electron pair from the nitrogen atoms in resonance. It is also accepted that the configuration of the dye molecules in the complex is *trans*. F. J. M. H. N. S.

1656. **Back-titration with mercuric nitrate in alkaline medium; potentiometric determination of aluminium and manganese.** H. Khalifa (Cairo Univ., Giza, Egypt). *Z. anal. Chem.*, 1958, **163** (2), 81–87.—After a brief discussion of other methods for the determination of aluminium and

manganese, a procedure is described which is simple, rapid and reasonably accurate in the range 10 to 100 μg for aluminium and 20 to 100 μg for manganese. A measured volume of standard EDTA (disodium salt) soln. is placed in a titration flask with 40 ml of buffer soln. (pH 8 to 11) and a known volume of the soln. under test. The mixture is then titrated with standard mercuric nitrate, the end-point being determined potentiometrically. A blank titration is then carried out. The difference between the end-points is a measure of the amount of EDTA soln. consumed by chelation with aluminium or manganese. A buffer soln. of pH 8 is recommended for the determination of aluminium, since at higher pH values errors occur owing to instability of aluminium - EDTA. Results are quoted for various concn. of the standard soln. and compared with those obtained by other methods. Cations which form chelate compounds with EDTA interfere. End-points may be determined within 0·01 to 0·02 ml of titrant with jumps of from 35 to 88 mV per 0·1 ml of titrant. S. M. MARSH

1657. **Determination of phosphorus in aluminium - silicon alloys.** M. V. Kuhn. *Fonderie*, 1958, (149), 279–282.—The metal is attacked by HCl in an atmosphere of free hydrogen. Soluble P is collected as phosphine in brominated HBr. The H_3PO_4 thus formed is determined photometrically by the reduced molybdophosphate method used in the analysis of steel. A significant proportion of P remains in the unattacked Si, which is therefore treated with a mixture of HNO_3 and HF. The P which remains in the residue after evaporation is determined in the same way as before. The coeff. of variation of results is $\simeq \pm$ 10%. The total P varied between 3 and 20 g per metric ton and was never as low as 1·5 g per metric ton, a figure frequently mentioned in literature on the subject. K. R. C.

1658. **Comparative study of photometric methods of determining gallium.** D. P. Shcherbov and A. I. Ivankova (Kazakh Inst. of Mineral Raw Materials). *Zavod. Lab.*, 1958, **24** (6), 667–674.—Photometric methods based on the use of 8-hydroxyquinoline, Rhodamine B, Rhodamine 6G, and gallion [8-amino-2-(3-chloro-2-hydroxy-5-nitrophenylazo)-1-naphthol-3; 6-disulphonic acid] are compared for sensitivity, selectivity, and concn. range of applicability. The most sensitive reagent and the one with the widest limits of application is Rhodamine 6G which, however, is less selective than Rhodamine S; 8-hydroxyquinoline is even less selective. The use of gallion, which has low selectivity, but fairly high sensitivity (0·05 μg per ml) and range of application, is recommended for the simplicity of its use when Ga can be separated first. G. S. SMITH

1659. **Determination of small amounts of indium in the presence of zinc.** P. K. Agasyan. *Nauch. Dokl. Vyssh. Shkolŷ Khim. i Khim. Tekhnol.*, 1958, (2), 308–313; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,531.—It is established that the potentiometric titration of In with $K_4Fe(CN)_6$ in the presence of Zn is not possible, because of the formation by both elements of ppt. of similar solubility, as a result of which the titration curve has only one sharp change of potential. To determine In in soln. containing Zn, first separate In with aq. NH_3, filter off the pptd. $In(OH)_3$, dissolve the ppt. in a small vol. of hot dil. HCl and, with an acidity 0·05 to 0·3 N in HCl or 2 to 4 N in acetic acid, titrate with a $K_4Fe(CN)_6$ soln. Determine the end-point of the titration potentiometrically. With a concn. of In $< 0{\cdot}0005\ M$ the error is $> \pm$ 1%. The ppt. formed corresponds to the formula $KIn_3[Fe(CN)_6]_4$; the solubility of this compound, determined radiometrically with the isotope ^{114}In, is $1{\cdot}94 \times 10^{-6}$ moles per litre. When determining smaller amounts of In (down to $6 \times 10^{-6}\ M$), first extract the In with a soln. of 8-hydroxyquinoline in $CHCl_3$ at pH 3·5 and then measure the extinction of the resulting extract at 395 mμ. The In can be determined in the presence of 25,000 times the amount of Zn. C. D. KOPKIN

1660. **Polarographic determination of indium in zinc-base alloys. IV. Determination of indium in mazak alloys.** G. F. Reynolds and H. I. Shalgosky (Min. of Supply, Chemical Insp., Woolwich, England). *Anal. Chim. Acta*, 1958, **19** (2), 190–194.—A previously described method (*Anal. Chim. Acta*, 1958, **18**, 607) is modified for alloys that contain Al. The Al and In are pptd. together as hydroxides at pH 4·95 and the ppt. is washed in the centrifuge. The ppt. occludes Cu and Cd, which are objectionable in the subsequent polarographic procedure, but, if the alloy is dissolved in 10 N HCl without the use of an oxidising agent, a negligible amount of Cu goes into soln. Cadmium is eliminated by a second pptn. of the hydroxides. By the use of ^{114}In it is shown that retention of In by the undissolved Cu and losses in the pptn. procedure cause an overall standard loss of 15%. The ppt. is dissolved in aq. HCl and the In determined polarographically with the use of a standard prepared from the same sample with the initial addition of a known wt. of In. The method is suitable for mazak alloys containing 0·5 to 20 p.p.m. of In. H. N. S.

1661. **Polarographic determination of indium.** D. Weiss (Res. Inst. Ores, Prague). *Hutn. Listy*, 1958, **13** (7), 641–643.—The determination is carried out in slightly acid chloride medium (pH 3). Several elements interfere, in particular Cd and V, the waves of which coincide with that of In. This interference can be avoided by masking V with NaF and removing Cd as a sol. ammoniate. The described method enables In to be determined in ores at concn. as low as 0·003%. J. ZÝKA

1662. **Spectrographic porous-cup technique for the analysis of indium alloys.** H. M. Hyman (Radio Corp. of America, Semiconductor Div., Somerville, N.J., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (3), 95–97.—The method described can be used to determine Ag (1·0 to 3·0%), Ga (0·1 to 2·0%), Zn (0·1 to 2·0%) and Al (0·1 to 1·0%) in indium alloys. The error does not exceed 4·0 to 8·3% of the amount found, depending on the element and its concn. K. A. PROCTOR

1663. **Titrations with quinol and analogous reducing agents. X. Titrations of cerium, chromium and vanadium and the possibility of their determination in presence of each other.** L. Mráz, V. Simon and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (6), 1089–1092.—The influence of acidity and vol. in the potentiometric titration of $Cr_2O_7^{2-}$, VO_3^- and Ce^{4+} with quinol has been studied. It was found that 0·1 N to 2×10^{-4} N $Cr_2O_7^{2-}$ can be titrated in 2 to 15% H_2SO_4; 0·1 N to 0·05 N VO_3^- in 15 to 30% H_2SO_4; 0·05 N to 0·005 N VO_3^- in 25 to 30% H_2SO_4 and 0·1 N to 5×10^{-4} N Ce^{4+} in 2 to 15% H_2SO_4; 0·1 N to 0·01 N $Cr_2O_7^{2-}$, VO_3^- and Ce^{4+} can

also be determined in 0·2 *N* to 15 *N* HNO_3. In a mixture of Ce^{4+} and VO_3^- (0·1 *N*) two sharp potential changes appear; another small change between $Ce^{IV} \rightarrow Ce^{III}$ and $V^V \rightarrow V^{IV}$ can be observed in the presence of $Cr_2O_7^{2-}$, but cannot be used for quant. evaluation. If Mn^{2+} are added to a mixture of $Cr_2O_7^{2-}$ and Ce^{4+}, Mn^{II} is oxidised to Mn^{IV}, and Ce^{IV} is reduced to Ce^{III}, whilst $Cr_2O_7^{2-}$ do not react; when titrating this mixture potentiometrically, a sharper potential change can be obtained than in the previous case. J. ZÝKA

1664. **Titrations with quinol and analogous reducing agents. XI. Determination of cerium in various materials.** L. Mráz, V. Simon and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (7), 1354–1356.—*Procedure for monazite*—Add conc. H_2SO_4 (20 ml) to the finely powdered sample (1 g) and heat for 5 hr. (add H_2SO_4 when necessary). Cool, dilute with H_2O to 250 ml and filter. To 10 ml of the filtrate add H_2SO_4 till 1 to 2 *N*, 0·1 *N* $AgNO_3$ (1 ml) and ammonium persulphate (5 g) and heat for 10 min. Cool and titrate potentiometrically or visually (to ferroin) with quinol soln. When determining Ce in cerite metals and alloys of Al - Th - Ce, dissolve the sample (0·2 to 0·3 g) in HNO_3 (1:1). A large excess of Pb, Al, Ni, Mo, Ag, Bi, As or Fe, and the presence of Hg, W, Sb, Rh or Pd cause errors; Cr, V and Mn must be absent. For the determination of a small amount of Ce in steel a new method is proposed; the excess of interfering elements is removed by electrolysis with a mercury cathode, Ce^{4+} are separated with $Al(OH)_3$, and the ppt. is dissolved and titrated. J. ZÝKA

1665. **Study of the flame emission characteristics of lanthanum in an aqueous-alcoholic medium.** O. Menis, T. C. Rains and J. A. Dean (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chim. Acta*, 1958, **19** (2), 179–189.—The flame spectrum of La is investigated with Beckman and ORNL recording spectrophotometers equipped with photomultiplier detecting devices. Data are presented for the five principal emission bands at 437 to 445 mμ, 538 to 568 mμ, 587 to 598 mμ, 701 to 772 mμ and 791 to 860 mμ. The addition of various alcohols to the test soln. enhances the emission of La and the use of soln. in 50% methanol is recommended to lessen interference from certain other metals. In general the interference from foreign ions is less at 743 mμ than at other wavelengths. In many types of sample such interference can be dealt with by the method of standard addition, and this method is used in the determination of La in commercial thorium and didymium oxides, glass (for pH electrodes) and mixtures of La_2O_3 with UO_2SO_4. H. N. S.

1666. **Zirconyl - alizarin blue S complex. A spectrophotometric study.** V. Gopalakrishna and B. S. V. Raghava Rao (Andhra Univ., Waltair, India). *Anal. Chim. Acta*, 1958, **19** (2), 161–165.—The blue lake formed by Zr^{4+} with alizarin blue S is used for the spectrophotometric determination of ZrO_2 in quantities down to 0·27μg. The lake is formed in 0·5 *N* HCl and the extinction measured at 625 mμ. Under these conditions the uncombined dye shows little absorption. The soln. of the lake is stabilised by the presence of 10% of acetone. Oxalate, tartrate, PO_4^{3-} and F^- must be absent, only trace quantities of Be^{2+} and Ti^{4+} are tolerated, and SO_4^{2-}, Th^{4+}, rare earths, Al^{3+}, U^{6+}, Ca^{2+} and Fe^{3+} should be present in only restricted amounts. H. N. S.

1667. **Detection of rare-earth ions as oxalates and cupferrates.** W. W. Wendlandt and D. W. Hayes (Texas Technol. Coll., Lubbock, U.S.A.). *Science*, 1957, **126**, 451–452.—The method of Irving *et al.* (*J. Chem. Soc.*, 1949, 1489) is modified to determine the sensitivity of the pptn. reaction of cupferron with rare-earth-metal ions. The pptg. soln. (0·1 *M*) (0·2 ml) is added to the rare-earth-metal chloride soln. (0·01 *M* or 0·001 *M*) to make a total vol. of 7 ml. The soln. is heated for 10 min. to 80°, and observed visually for a ppt. after cooling. Cupferron is a more sensitive reagent than oxalic acid for the detection of rare-earth ions. The reaction sensitivity, in μg of Mn^{2+} per ml for oxalic acid and cupferron, respectively, was La, 6·3, 2·4; Ce, 6·4, 1·6; Pr, 6·4, 1·6; Nd, 6·6, 2·5; Sm, 6·9, 5·1; Gd, 17·8, 17·8; Y, 10·2, 10·2. K. R. C.

1668. **Use of copper as a cation exchanger in the separation of rare-earth elements by ion exchange using ethylenediaminetetra-acetic acid.** J. Loriers and C. Lenoir. *Compt. Rend.*, 1958, **247** (4), 468–471.—Rare-earth elements in soln., complexed with EDTA, can be fixed selectively on a resin saturated with Cu^{2+}. Effective separations are obtained with EDTA by means of a ring of cations (Cu^{2+}) in the columns. The soln. containing the salt formed by copper and EDTA can be used as eluents. K. R. C.

1669. **Analysis of rare-earth elements. II. The absorption spectra of lanthanide elements in the near infra-red region.** Giichi Muto and Masato Mamiya (Inst. of Ind. Sci., Tokyo Univ., Yayoi-cho, Chiba). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (7), 809–812.—Although La, Ce^{4+}, Pr and Nd show no absorption at 1000 to 1200 mμ, Sm gives a distinctive absorption max. at 1090 mμ. The extinction is proportional to concn. of Sm of < 6 g per 100 ml in a chloride soln. The absorption band is not affected by other rare-earth elements of the cerium group, and its use appears to be far superior to that at 405 mμ for the determination of Sm. K. SAITO

1670. **Apparatus for the continuous determination by conductimetry of traces of carbon monoxide in gases.** J. Waclawik (Inst. de Chim. Générale, Warsaw, Poland). *Chim. Anal.*, 1958, **40** (7), 247–249.—An apparatus is described in which the gas, after passage through pressure-regulators, is purified and dried by passage through silica gel, ascarite, silica gel with sulphur trioxide, fuming H_2SO_4, and solid KOH. These remove water, acid and basic components, and unsaturated hydrocarbons. A predetermined proportion of the gas then passes through heated I_2O_5, which oxidises the CO to CO_2, then through heated silver wool, which removes iodine formed by the oxidation. The gas then meets a stream of NaOH soln. (concn. not stated), which has passed through a conductivity cell, and which absorbs the CO_2. After the absorption the conductivity is again determined in a second cell, the loss of conductivity measuring the CO_2. The electrolyte then passes to a special electrolytic cell, where it is regenerated before recirculation. In the apparatus is incorporated another electrolytic cell, in which CO_2 for calibration of the recording system is obtained by electrolysis of saturated oxalic acid soln., the amount of CO_2 being regulated by control of the electrolysing current. R. E. ESSERY

1671. **Analysis of nickel alloys for oxide cathodes. VI. Determination of silicon.** G. Eckert (Wernerwerk für Bauelemente, Siemens & Halske A.-G., Munich, Germany). *Z. anal. Chem.*, 1958, **162** (6), 408–413.—Silicon is determined photometrically as the reduction product of molybdosilicic acid at 730 mμ, a blank containing an equal amount of pure Ni being necessary. If the nickel alloy contains W, then the tungstic acid which separates should be removed by centrifuging. The coeff. of variation is ± 2·5%, but increases to ± 5% in the presence of W. The method appears to be suitable for the determination of Si in other metals and alloys.

J. H. WATON

1672. **Analysis of pure metals. Determination of silicon impurity in certain semi-conducting metals.** V. A. Nazarenko and G. V. Flyantikova (Inst. of Gen. and Inorg. Chem., Acad. Sci. UkrSSR). *Zavod. Lab.*, 1958, **24** (6), 663–666.—Since the formation of the complex molybdosilicic acid is incomplete at the acidities necessary for retaining Sb, Ga and In in soln., and the separation of silica is impracticable for the determination of traces of Si in the pure metals, the removal of the metal is necessary. Use is made of the volatility of $SbBr_3$, of the volatility of the 8-hydroxyquinoline complex of Ga which is appreciable at 100°, of the volatility of $InCl_3$ (complete at 600°), and of the volatilisation of Tl_2O when salts of Tl are fused with NaOH (Kuznetsov and Myasoedova, *Anal. Abstr.*, 1957, **4**, 2132). For prevention of the interference of P, As and Ge in the molybdosilicate method for Si, the use of citric acid, which is added after the formation of the complex, is effective. *Determination of Si in antimony*—The powdered sample (0·5 g or, if the content exceeds 0·0004%, 0·25 g) in a platinum dish is dissolved in 5 ml of conc. HNO_3, the soln. is evaporated to dryness, oxides of N are removed by heating, the residue is treated with 5 ml of 8 to 9 *N* HBr, the soln. is evaporated to dryness, a further 2·5 ml of HBr is added, and the soln. is again evaporated to dryness. The residue is then treated with 0·15 ml of 1·5 *N* NaOH, the water is evaporated off and the residue is fused for 5 min. The cooled melt is treated with 0·5 ml of *N* H_2SO_4 and 0·5 ml of water, the soln. is heated and then cooled and passed through a paper in a waxed funnel, the filter being washed with water (2 × 1 ml) so that the total filtrate is 2·8 ml. The soln. is treated with 0·2 ml of 10% $(NH_4)_2MoO_4$ soln., and several standards (0 to 4 μg of Si in 2·8 ml) are treated with the same vol. of reagent. All the soln. are kept at 100° for 5 min. and then treated with 1 ml of 50% citric acid soln. and again heated for 5 min. The cooled soln. are mixed with 1 ml of 10 *N* H_2SO_4 and then with 0·2 ml of a 0·5% soln. of $SnCl_2$ in 2 *N* HCl, the complex is extracted with 2 ml of *iso*amyl alcohol and the colours are compared visually or measured with a photometer. *Determination of Si in gallium*—The sample (0·5 g) is dissolved in a platinum dish in 5 ml of 10 *N* HCl, and the soln. is mixed with 2 ml of 10 *N* HCl and 10 ml of a 15% soln. of 8-hydroxyquinoline in ethanol and evaporated to dryness on a boiling-water bath. The residue is carefully ignited and then moistened with 2 ml of HCl, and the soln. is again evaporated and the residue is ignited. Treatment with HCl, etc., is carried out two or three more times. The residue is treated with 0·5 ml of 2 *N* NaOH and 2 ml of water, the soln. is evaporated to dryness, and the residue is fused for 5 min. The cooled melt is dissolved in 5 ml of water and the soln. is neutralised to bromocresol purple paper with 2 *N* H_2SO_4 (0·5 ml), and 1 ml of acid in excess is added. The soln. in a 25-ml flask is treated with 1 ml of 10% $(NH_4)_2MoO_4$ soln., heated at 100° for 5 min., mixed with 2 ml of 50% citric acid soln., again heated for 5 min., then cooled, mixed with 3 ml of 10 *N* H_2SO_4, and extracted with *iso*amyl alcohol (2 × 2 ml). The extracts are shaken with 1 ml of 2 *N* H_2SO_4 and 0·2 ml of a 0·5% soln. of $SnCl_2$ in 2 *N* HCl, and the extinction of the separated *iso*amyl alcohol layer is measured with a red filter. *Determination of Si in indium*—The sample (0·5 g) is dissolved in 2 ml of 10 *N* HCl, the soln. is evaporated and the residue is ignited at 400° to 450°. Further treatment with 10 *N* HCl (1 ml each time) and evaporation is carried out four or five times until there is no visible residue. Treatment with 0·5 ml of 2 *N* NaOH, etc., is carried out as with gallium. *Determination of Si in thallium*—The sample (0·5 g) is dissolved in 1 ml of conc. HNO_3, the soln. is evaporated to dryness, the residue is dissolved in 5 ml of water, and the soln. after addition of 0·5 ml of 85% formic acid soln. is evaporated to dryness, avoiding fusion. The residue is dissolved in 2 ml of water, 1 ml of 2 *N* NaOH is added, and the soln. is evaporated to dryness and the residue fused to give a reddish-brown melt, which after addition of 0·5 ml of water and evaporating is heated over a burner for 5 to 10 min. until white fumes cease to be evolved. Treatment with 0·5 ml of water, etc., is carried out three or four more times, and the removal of Tl is considered to be complete when the residue is a white melt of NaOH. The cooled melt is dissolved in 5 ml of water and the soln. is neutralised (bromocresol purple paper) with 2 *N* H_2SO_4 (1 ml) and 1 ml in excess is added. The soln. in a 25-ml flask is treated with 1 ml of 10% $(NH_4)_2MoO_4$ soln., etc., as described for gallium. G. S. SMITH

1673. **Separation of germanium by paper chromatography. Evaluation of chromatograms.** Z. Nagy and E. N. Pólyik (Inst. Med. Chem., Med. Univ., Debrecen, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1958, **16** (1), 9–12 (in English).—A solvent mixture containing 20 ml of $CHCl_3$, 4·5 ml of ethanol and 0·5 ml of HCl has been found suitable for the separation of Ge^{4+} from 20 other cations. The spots were detected by u.v. fluorescence after treatment with a 0·3% ethanolic solution of morin. As little as 0·1 μg of Ge^{4+} may be detected.

T. R. ANDREW

1674. **Determination of lead with mercaptobenzothiazole in the presence of barium.** V. Krajovan-Marjanović, R. Podhorsky, S. Žilić and D. J. Bačić (Anal. Chem. Lab., Fac. Technol., Univ. Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (2), 135–139.—The method of Spacu and Kuraš for the determination of Pb by pptn. with mercaptobenzothiazole (*Z. anal. Chem.*, 1936, **104**, 88) has been applied to the determination of Pb in the presence of larger quantities of Ba. Separation was successful when an aq. NH_3 - NH_4NO_3 buffer was used as the pptn. medium, and it was shown that the Ba did not interfere unless the Pb to Ba ratio was less than 1:100. It was found that the time of standing before filtration, the temp. of the soln. during filtration and the dilution of the soln. up to ten times with water have no influence on the results.

H. M.

1675. **Spectrographic analysis of specially pure tin by means of preliminary enrichment.** D. M. Shvarts and V. V. Portnova (State Sci. Res. and Development Inst. "Gipronikel"). *Zavod. Lab.*,

1958, **24** (6), 731–734.—The sample under a layer of CCl_4 is chlorinated by passage of a current of Cl, and the impurities are extracted by adsorption on activated carbon, which is then analysed spectrographically. G. S. SMITH

1676. **The use of naphthalene derivatives in inorganic analysis. V. Further applications of the nitroaminonaphthalenemonosulphonic acids to the fluorimetric detection of stannous tin.** J. R. A. Anderson, B. Crawford and J. L. Garnett (N.S.W. Univ. of Technol., Sydney, Australia). *Anal. Chim. Acta*, 1958, **19** (1), 1–4.—The influence of the sulphonic acid grouping was studied by carrying out the stannous tin spot-test with the ammonium salts of 5-nitro-1-naphthylamine-2-sulphonic acid (**I**), 4-nitro-1-naphthylamine-2-sulphonic acid (**II**) and 4-nitro-1-naphthylamine-6-sulphonic acid (**III**). Spots (0·01 ml) containing 0·1 mg of metal ion were placed on Whatman filter-paper and sprayed with a 0·1% soln. of the reagent, and any fluorescence under u.v. light before and after spraying with 15 *N* aq. NH_3 was noted. Of the 47 metal ions tested, only stannous tin gave an intense fluorescence under u.v. light after quenching with aq. NH_3. The sensitivity of the method was—**I** 10^{-5}, **II** and **III** 10^{-6} g of Sn^{II}. F. J. M.

1677. **Polarographic studies with fused borax as basal solution. II. Polarography of tin, antimony and bismuth.** K. M. Kalabalina and Yu. K. Delimarskii. *Ukr. Khim. Zhur.*, 1958, **24** (2), 152–157; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,502.—The polarographic behaviour of SnO_2, Sb_2O_3 and Bi_2O_3 in a basal soln. of fused $Na_2B_4O_7$ was studied and polarograms were recorded at 820°. It was established that for all three compounds there is a direct proportionality between the value of the diffusion current and molar concn. (*M*). All the polarograms obtained were better satisfied by the Heyrovský - Ilkovic equation than by the Kolthoff - Lingane equation. The $E_{\frac{1}{4}}$ was not a linear function of log *M*/2. In the case of SnO_2, $E_{\frac{1}{4}}$ is const. to the first approximation and is −0·760 V; for Sb_2O_3 the greatest deviation from the average value (−0·382 V) was 0·022 V; for Bi_2O_3, $E_{\frac{1}{4}}$ is −0·800 V at *M* = 0·001 and moves towards more positive potentials with increase of *M*, but not with a linear relationship. It is observed that the polarographic processes correspond to the conversion of SnO_2 into SnO, and of Sb_2O_3 into Sb_2O_4 or Sb_2O_5. (For Part I, *cf. Anal. Abstr.*, 1958, **5**, 4003.) C. D. KOPKIN

1678. **Use of hydroxyflavones in analytical chemistry. Photometric determination of titanium with quercetin.** A. P. Golovina, I. P. Alimarin and N. F. Stepanov. *Nauch. Dokl. Vŷssh. Shkolŷ Khim. i Khim. Tekhnol.*, 1958, (2), 285–289; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,538.—The reaction of quercetin (**I**) with several elements was studied. It is established that with Ti^{4+} **I** forms an intensely coloured complex with max. light absorption at 425 mμ. At pH 3·3 to 6·0 the intensity of the colour is practically const.; at pH < 1·0 the complex is not formed. To stabilise the coloured soln. the addition of 20% by vol. of ethanol or acetone is recommended. The intensity of the colour reaches a max. 20 min. after mixing the soln. if ethanol is present, and after 1 hr. if acetone is present; the intensity is then const. for 4 to 6 hr. The optical density of the coloured soln. is independent of temp. in the range 20° to 70°. The complex contains Ti and **I** in the ratio 1:1. In determining Ti photometrically it is recommended the optical density be measured at 440 to 450 mμ; in this range the extinction of **I** itself is practically absent. Beer's law is obeyed for concn. of Ti of 0·05 to 1 μg per ml. C. D. KOPKIN

1679. **Determination of micro amounts of titanium in a niobium base.** Yu. A. Chernikhov, Sh. G. Melamed and B. M. Dobkina (State Inst. of Rare Metals). *Zavod. Lab.*, 1958, **24** (6), 677–679.—When the ratio of the concn. of Nb and Ti exceeds 100:1 the interference of Nb in the H_2O_2 colorimetric method for Ti is serious. If the intensity is measured at 470 to 480 mμ the interference is practically eliminated, but the sensitivity of the determination is too low. A spectrographic method (d.c. arc, 10 amp.) sensitive to 0·002 % of Ti is described. The sample (10 mg of Nb_2O_5 containing Ti) is mixed with the same amount of carbon powder and evaporated from a carbon electrode. After 2·5 min. the whole of the Ti present has passed into the arc. The lines Ti 3234·5 A and Nb 3244·5 A are measured. Standards are prepared by mixing pure Nb_2O_5 and TiO_2. The reproducibility of the method is 15%. G. S. SMITH

1680. **Photometric determination of titanium and its separation from iron, chromium, nickel and other elements by means of the strong basic anion exchanger Amberlite IRA-400.** J. Korkisch and A. Farag (Chem. Inst. II, Univ. Vienna). *Mikrochim. Acta*, 1958, (5), 659–673.—In a Na acetate soln. in the pH range 3·6 to 4·0 the addition of ascorbic acid soln. of similar pH forms with Ti an intensely yellow and stable complex which can be measured between 350 and 365 mμ. As the ascorbate complexes of U, V and Mo absorb in this wavelength range and as the ions Cr^{3+}, Ni and Fe^{3+}, because of their own colours, interfere to give high values for Ti, these elements must, when present in sufficient quantity to exceed the measuring error of ± 6%, be separated from the Ti. Such elements as Fe, Cr and Ni are separated by an ion-exchange operation, based on the fact that the Ti complex is held by Amberlite IRA-400 (ascorbate form). The method described is of general application for microgram as well as milligram amounts of Ti, except for minerals containing Cu, U, V, Mo and W and also phosphates. D. F. PHILLIPS

1681. **Separation of zirconium from chromium and aluminium by ion-exchange chromatography.** T. A. Belyavskaya and M. K. Chmutova. *Nauch. Dokl. Vŷssh. Shkolŷ Khim. i Khim. Tekhnol.*, 1958, (2), 305–307; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,495.—The adsorption of Zr, Cr and Al on ion-exchange resins from 0·1 to 6·0 *N* HCl is studied. Anion-exchange resins (EDE-10 and AN-2F) (Cl form) do not adsorb Zr, Cr and Al at any concn. of HCl. The cationite KU-2 (H form) in dil. HCl (0·1 to 1·0 *N*) adsorbs Zr completely; the adsorption of Cr under these conditions is incomplete and decreases with increase of concn. of HCl; Al is completely adsorbed from 0·1 *N* HCl, while it is practically not adsorbed at all from *N* HCl. On increasing the concn. of HCl to 4 or 6 *N* there is no adsorption of the three elements. A chromatographic method of separating Zr from Cr and Al is proposed, based on their different adsorptions on KU-2 from *N* HCl. Fill a column (height 11 cm, diam. 1 cm) with the cationite KU-2 (H form), wash it with *N* HCl, introduce the soln. of acidity *N* in HCl, and wash the column with *N* HCl till Cr and Al are completely extracted. Elute the Zr with 4 *N* HCl. C. D. KOPKIN

1682. **Co-precipitation in quantitative analysis. V. Effect of Complexone on the precipitation of zirconium phosphate.** A. K. Babko and M. I. Shtokalo (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1958, **24** (6), 674–677.—The pptn. of Zr^{4+} by Na_2HPO_4 in the presence of EDTA yields a slowly forming cryst. ppt. instead of the usual amorphous ppt. and the co-pptn. of other ions is reduced. The acidity necessary for destroying the EDTA complex of Zr is 3 to 4 *N*. This observation is applied to the separation of Zr and Ti. A soln. (5 ml of 0·1 *M*) of the chlorides of Zr and Ti was mixed with 2 ml of conc. HCl, 3 ml of 30% H_2O_2 and 5 ml of 0·1 *M* EDTA, and diluted to 100 ml. The soln. was then treated with 36 ml of 10% Na_2HPO_4 soln. and dropwise with 19 ml of dil. H_2SO_4 (1:1) and set aside overnight. The Ti in the ppt. was determined (0·18% of the quantity originally taken) and compared with the amount of Ti in ppt. obtained in the usual manner without the use of EDTA (2·1% of the amount taken).
G. S. Smith

1683. **The chromatography of thorium.** G. Almássy, M. Ördögh and B. Hadobás (Chem. Dept., Experimental Atomic Reactor of the Central Phys. Res. Inst. of the Hungarian Acad. of Sci., Budapest). *Magyar Kém. Foly.*, 1958, **64** (7–8), 240–241.—The chromatographic separation of Th on paper or on a cellulose column was re-examined; the solvent was anhyd. diethyl ether - HNO_3, as recommended by Kember. By using empirical equations it was proved that the discontinuity of the spots or bands, due to the action of the anhyd. solvent, is caused by a Freundlich isotherm. The authors found that increasing the acid or H_2O content, or both, of such a solvent will remedy this. The best solvent is prepared by shaking 8 *N* HNO_3 (100 ml) and ether (100 ml) for 10 min.; the upper (organic) layer is used. Thorium has an R_F value of 0·9; that of most other metals is zero. Since 100 ml of solvent is sufficient for the removal of 200 mg of ThO_2 from the column, the method can be used for quant. determinations. A. G. Peto

1684. **Determination of thorium by precipitation with sodium benzenesulphinate.** I. P. Alimarin and S. S. Alikberov (M. V. Lomonosov Moscow Inst. of Fine Chemical Technology). *Zavod. Lab.*, 1958, **24** (7), 804–807.—The white cryst. ppt. of $Th(C_6H_5SO_2)_4$ obtained from 0·5 *N* HCl soln. is dried at 110° and weighed. Most elements, including Be, Al, and rare earths and small amounts of Ti and Fe do not interfere. With large amounts of Fe, interference is prevented by reduction with ascorbic acid and complexing the Fe with EDTA. Uranyl does not interfere, but U^{4+} give a green ppt. that can be used for the determination of U. Interference is caused by Zr, but Zr can be separated by pptn. in more acid medium. G. S. Smith

1685. **New method, based on chromatography, for the estimation of thorium and uranium in monazite.** I. I. M. Elbeih and M. A. Abou-Elnaga (Univ. of Cairo, Egypt). *Anal. Chim. Acta*, 1958, **19** (2), 123–128.—The sample of monazite sand is treated with conc. H_2SO_4, the extracted metals are pptd. as oxides with NaOH and the oxides are dissolved in 50% HNO_3. The Th and U are separated from other metals by paper-strip chromatography, the solvent consisting of dioxan (100 ml), conc. HNO_3 (1 ml), phenazone (1 g) and water (2·5 ml). The Th and U spots are too close for separate treatment. Both are extracted together with 0·001 *M* EDTA acidified with 0·1 *N* HNO_3, a measured vol. of 0·001 *M* $Bi(NO_3)_3$ is added and the excess is titrated with 0·001 *M* EDTA, catechol violet being used as indicator. The total 0·001 *M* EDTA consumed minus the vol. of 0·001 *M* $Bi(NO_3)_3$ is a measure of the Th present. From further chromatograms the uranium zones, containing some Th, are extracted with ethanol after soaking in ethanol containing a little HNO_3. The acidity is removed with Na_2CO_3 soln., the ethanol is evaporated and the aq. soln. is treated with Na_2CO_3 and H_2O_2. The pptd. thorium dioxide is filtered off and the U is determined spectrophotometrically as peroxide in the filtrate after further additions of Na_2CO_3 and H_2O_2.
H. N. S.

1686. **Field sampling and laboratory assay of anhydrous ammonia.** W. J. Ingram (State Dept. Agric., Salem, Oreg., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 626–629.—About 6·7 g of anhydrous NH_3, weighed in a 10-ml tube, with correction for the weight of air contained in the empty tube, is passed into *N* H_2SO_4 standardised with elimination of CO_2 error. The excess of acid is titrated with standard alkali. Commercial samples of ammonia contained 82·09 to 82·17% of N.
A. A. Eldridge

1687. **Simultaneous ultra-violet spectrophotometric determination of nitrate and nitrite.** Hiroshi Hamaguchi, Rokuro Kuroda and Shinya Endo (Chem. Dept., Fac. of Sci., Tokyo Univ. of Educ., Koishikawa, Tokyo). *Japan Analyst*, 1958, **7** (7), 409–415.—The u.v. absorption of NO_3^- and NO_2^- is sensitive to change in pH, but provides distinctive maxima at 302 and 355 mμ, respectively, in a neutral soln. of pH > 5. The peak height at 355 mμ is little affected by varying the concn. of NO_3^-, whilst that at 302 mμ is affected by the concn. of NO_2^-; nevertheless these two ions can be simultaneously determined with these two key bands. The working curve is linear for < 4 mg of NO_3^- (355 mμ) and < 15 mg of NO_2^- (302 mμ, corrected) per ml. No interference results from free alkali other than aq. NH_3, which must be approx. neutralised with HCl before the determination. Free acids are neutralised with NaOH. Ordinary colourless cations (except Al^{3+}, which must be converted into $NaAlO_2$) and nitrogen (in various forms other than hydrazine) are harmless. Oxalate and S^{2-} increase the value for NO_3^-. K. Saito

1688. **Determination of phosphoric acid in superphosphates by means of ion exchangers.** I. G. Lakomkin (Lensovet Leningrad Tech. Inst.). *Zavod. Lab.*, 1958, **24** (6), 679–680.—The sample (2·5 g) is boiled with 50 ml of 10% HCl soln., the cooled soln. is diluted to 250 ml and 100 ml of the clear soln. after filtration is neutralised by addition of 2 *N* NaOH followed by 2 *N* HCl, then diluted to 250 ml and passed through a column of cationite (SBS) (Na form), which is then washed with water until 500 ml is collected. An aliquot (100 ml) is used for the determination of H_2SO_4 by the benzidine volumetric method. A second aliquot (100 ml) is treated with 3 g of NaCl, 1 to 2 drops of methyl orange indicator, and 2 *N* NaOH to give a yellow colour. The soln. is then titrated with 0·1 *N* NaOH until the colour is pale pink to phenolphthalein indicator. After addition of 10 to 15 ml of *N* $Pb(NO_3)_2$ to react with HPO_4^{2-}, forming $Pb_3(PO_4)_2$ and liberating free acid, the soln. is further titrated. The concn. of H_3PO_4 is calculated from the titration of the third H ion. G. S. Smith

1689. **Analysis of solutions containing mixtures of linear sodium phosphates.** A. G. Buyers (Colgate Palmolive Co., Jersey City, N.J., U.S.A.). *Anal. Chim. Acta*, 1958, **19** (2), 118–122.—The method is applicable to mixtures of sodium ortho-, pyro- and tri-phosphates. Pyrophosphate is determined by double pptn. with $ZnSO_4$ at pH 4·0 to 4·5 and at 19°, the $Zn_2P_2O_7$ being weighed after ignition at 900° to 940°. A standard soln. of $AgNO_3$ is titrated conductimetrically with a soln. of the sample. The total PO_4^{3-} after hydrolysis is determined by pptn. as $Mg(NH_4)PO_4.6H_2O$ and ignition to $Mg_2P_2O_7$. From the results of these three determinations the amount of each acid radical is calculated.
H. N. S.

1690. **Solvent extraction of trace antimony(V).** G. K. Schweitzer and L. E. Storms (Univ. of Tennessee, Knoxville, Tenn., U.S.A.). *Anal. Chim. Acta*, 1958, **19** (2), 154–161.—In a study of the extraction of Sb^{5+} from aq. HCl with diethyl, di*iso*propyl, di*iso*amyl and di-*n*-hexyl ethers and chloroform, ^{124}Sb has been used. Mutual solubility of the phases and the distribution of HCl between them are determined with the original concn. of HCl varying from 0 to 12 *N*. Salting-out effects are investigated with diethyl ether as extractant. Spectrophotometric evidence is obtained for the existence of chloro and chlorohydroxo complexes of antimony and the dielectric constants of the ethereal phases are determined. The relation of all these factors to the distribution of antimony between the phases is discussed and di*iso*propyl ether is found to be the most effective extractant.
H. N. S.

1691. **Polarographic analysis of "standfast" alloy.** S. H. Ibrahim and A. P. Madhavan Nair (A.C. College of Tech., Madras Univ., India). *J. Sci. Ind. Res., B, India*, 1958, **17** (5), 190–191.—A polarographic method is described for the rapid determination of low-melting alloys such as "standfast" alloy. Bismuth, Pb and Cd in the alloy can be determined directly in 40 to 50 min. A soln. of the alloy in *M* HNO_3, after pptn. of Sn as metastannic acid, gives well-defined waves for Bi at − 0·01 V, for Pb at − 0·13 V and for Cd at − 0·59 V *vs.* the S.C.E. By comparing the diffusion currents of these metals in the test soln. with independent calibration curves of the respective metals, it is possible to determine accurately the three metals in the alloy. The amount of Sn present is determined by difference.
I. Jones

1692. **Simultaneous polarographic determination of bismuth and antimony in anode copper.** Yukiharu Yamazaki (Gov. Ind. Chem. Res. Inst., Shibuya-ku, Tokyo). *Rep. Gov. Ind. Chem. Res. Inst., Tokyo*, 1958, **53** (6), 213–216.—Bismuth (0·005 to 0·02%) and Sb (0·01 to 0·15%) in anode copper are co-pptd. with MnO_2 and polarographically determined in *N* H_2SO_4 containing 0·1 *N* HCl and 0·01% gelatin. The wave height increases with increase in concn. of H_2SO_4 and HCl and remains unchanged for 0·5 to 1 *N* H_2SO_4 and 0·1 to 0·3 *N* HCl, being proportional to the concn. of Bi and Sb for 0·05 to 2 mg each per 25 ml at −0·11 and − 0·23 V *vs.* the S.C.E., respectively. The polarogram is not affected by < 2 g of $MnSO_4$, or < 1 mg of Pb and Fe^{2+}. *Procedure*—Dissolve the sample (5 to 10 g) in dil HNO_3 (1:2, 150 ml), partly neutralise with aq. NH_3 soln. to make the acid concn. 0·3 to 1 *N*, dilute to 300 ml and heat with *N* $KMnO_4$ (5 ml) and $MnSO_4$ soln. (20%, 10 ml). Add *N* $KMnO_4$ (5 ml) and $MnSO_4$ soln. (20%, 10 ml) to the filtrate, dissolve the two portions of MnO_2 in acid H_2O_2 and repeat the pptn. 3 times. Dissolve the final ppt. in 6 *N* H_2SO_4 and an excess of 3% H_2O_2, heat to white fumes, add gelatin and dilute to 50 ml with HCl and record the polarogram.
K. Saito

1693. **Analysis of pure metals. Determination of traces of cobalt in bismuth.** V. A. Nazarenko and G. G. Shitareva (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1958, **24** (8), 932–934.—Cobalt (0·1 to 0·3 μg) in the presence of Bi (1 g) is determined by addition of 1-nitroso-2-naphthol to the slightly alkaline citrate soln., extraction of the cobalt complex with $CHCl_3$, successive evaporations of the extract with HNO_3 and $HClO_4$, ignition of the residue, dissolution in dil. HNO_3, and colorimetric determination as the nitroso-R salt.
G. S. Smith

1694. **The determination of small amounts of vanadium.** R. Bock and S. Gorbach (Farbwerke Hoechst A.-G., Frankfurt/M-Höchst, Germany). *Mikrochim. Acta*, 1958, (5), 593–629.—There is no universal method which is applicable to the determination of trace amounts of V in any given material. But by suitable combination, according to the nature of the sample being analysed, of methods for the decomposition, separation and finish, it is possible to determine V down to $\simeq 10^{-4}$% and under favourable conditions down to $\simeq 10^{-7}$% in most materials. Electrolysis on the mercury cathode and extraction of V oxinate, cupferrate or dithiocarbamate seem to be especially suitable for the separation. Polarography in weak alkaline borate buffer is preferred for the final determination because of low interference and high sensitivity.
D. F. Phillips

1695. **The behaviour of the complexes of vanadium, molybdenum and tungsten with ascorbic acid on the strong basic anion exchanger Amberlite IRA-400. Separation of vanadium from molybdenum, tungsten, iron and other elements.** J Korkisch and A. Farag (Chem. Inst. II, Univ., Vienna). *Mikrochim. Acta*, 1958, (5), 646–658.—In ascorbic acid soln. at pH 4 the ascorbate complexes of Mo, W and V which are negatively charged have diverse stabilities and are held by the exchanger with various degrees of firmness. Elution of V with 0·1 *N* HCl is 100% quantitative, of W only 1%, and Mo is not removed at all. Since Fe, Cr, Mn, etc., do not interfere in this pH range, the method may be used for the determination of V in steel. The mean error on three steel samples covering the range 0·2 to 0·6% of V was ± 6·2%, of which ± 5% was ascribed to the imprecision of the photometric measurement.
D. F. Phillips

1696. **Activation analysis of vanadium in high-alloy steels using manganese as internal standard.** G. Leliaert, J. Hoste and J. Eeckhaut (Lab. for Anal. Chem., Univ. of Ghent, Belgium). *Anal. Chim. Acta*, 1958, **19** (1), 100–107 (in English).—In the radio-activation method developed, manganese present in the steel sample is used as an internal standard, thus permitting accurate correction of neutron flux irregularities between sample and reference. The method was tested on 4 National Bureau of Standards samples having a vanadium content of 0·014 to 2·04% and a widely different composition as regards other alloy constituents. The results obtained by radio-activation in the Belgian Reactor BR1 and the Amsterdam 28 MeV deuteron cyclotron show satisfactory accuracy as well as precision.
F. J. M.

1697. **Spectrophotometric determination of niobium in alloys.** I. P. Kharlamov, P. Ya. Yakovlev and M. I. Lȳkova (Central Sci. Res. Inst. of Ferrous Metallurgy). *Zavod. Lab.*, 1958, **24** (8), 928–932.—The soln. obtained by fusing Nb_2O_5 with K_2CO_3 and extracting the melt with water has a max. absorption at 234·5 mμ and obeys the Beer-Lambert law within the concn. range of 5 to 25 μg per ml. To determine Nb (0·5 to 3%) in alloys of iron, chromium, cobalt or nickel containing Si, W, Mo, etc., the sample is boiled with 60 ml of dil. HCl (1:1), the soln. is oxidised with 3 to 5 ml of conc. HNO_3 and evaporated to dryness, and the residue is heated for 1 hr. with 40 to 50 ml of dil. HCl (1:4). After addition of 150 ml of hot water and some filter-paper pulp the soln. is boiled and then kept hot for 1 or 2 hr. (preferably overnight). The ppt. is collected and washed with *N* HCl until it is free from Fe, then ignited and fused with 2 g of K_2CO_3 - Na_2CO_3 mixture at 850° to 900°. The melt is extracted with 80 to 100 ml of cold water, the filtered soln. is diluted to 250 ml and the extinction is measured, a 10-mm quartz cell being used. Contents of Ti ≯ 1·5% give practically no interference. G. S. Smith

1698. **Separation of niobium and tantalum with cupferron.** A. K. Majumdar and J. B. Ray Chowdhury (Jadavpur University, Calcutta, India). *Anal. Chim. Acta*, 1958, **19** (1), 18–22 (in English).—Niobium is completely pptd. by cupferron from 1:1 mixtures of Nb and Ta in tartrate soln. buffered with ammonium acetate - acetic acid to a pH between 4·5 and 5·5, while Ta remains in soln. Higher concn. of Ta tend to keep the Nb in soln. but, with magnesia as coagulating agent, 1:2 mixtures can be resolved when Nb is co-pptd. with Ti; the maximum ratio for complete separation is 1:3. By co-pptg. Nb with Sn^{2+} or Sn^{4+}, mixtures of 1:30 and 30:1 can be satisfactorily separated. The addition of EDTA and tartaric acid inhibits interference from a wide range of ions, with the exception of U, Be, Ti and PO_4^{3-}; Fe^{III} and other tervalent elements in 100-fold excess require double pptn. F. J. M.

1699. **Separation of niobium and tantalum with N-benzoyl-N-phenylhydroxylamine.** A. K. Majumdar and A. K. Mukherjee (Jadavpur University, Calcutta, India). *Anal. Chim. Acta*, 1958, **19** (1), 23–26 (in English).—Tantalum is pptd. by N-benzoyl-N-phenylhydroxylamine at pH values below 1·5 and Nb from 3·5 to 6·5. In this pH range Nb is completely pptd. while Ta remains in soln.; Ta can then be pptd. by lowering the pH. A single pptn. separates mixtures of Nb and Ta in ratios of 1:16 and 100:1. Effective separation at ratios of 1:100 can be achieved by double pptn. The addition of EDTA inhibits interference from a wide range of ions including those of the rare-earth elements, but titanium, zirconium, vanadate and molybdate interfere. F. J. M.

1700. **The polarographic determination of oxygen and carbon dioxide.** C. Perovici and D. Dimitriu. *Rev. Chim., Bucharest*, 1958, **9** (6), 344–346.—It is shown that the differences between polarographic curves of soln. of O and CO_2 in aq. tetramethylammonium iodide (0·1 *M*) in an atmosphere of N are functions of the concn. of O and CO_2. The position of the half-wave potential varies with the concn. of O and, with high percentages, the wave is displaced by − 0·15 V, while the displacement of the curve up to + 0·45 V is a direct measure of the concn. of CO_2. It is claimed that mixtures of oxygen and up to 20% of CO_2 can be analysed. H. Sher

1701. **Separation and micro-determination of carbon dioxide and sulphur dioxide by means of detector tubes.** Yoshitaka Kobayashi (Fac. of Engng, Yokohama Univ., Minami-ku). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (6), 676–678.—When air (100 ml) containing < 3% of SO_2 and < 0·6% of CO_2 is passed at 100 ml per 200 sec. through a detector tube containing activated alumina impregnated with alkaline thymolphthalein (*Anal. Abstr.*, 1959, **6**, 1946) a colourless layer is produced near the gas inlet, the length varying with the concn. of SO_2 (0·05 to 3%) which is determined by means of an empirical curve at a given temp. No mutual interference was observed for SO_2 and CO_2. Chlorine, NO_2, ClO_2 and acetic acid interfere. Sulphur dioxide (< 0·2%) is also determined by a detector tube (diam. 2 mm) containing silica gel (40 to 60 mesh, 0·20 g) (water content 20%) impregnated with KIO_3 (0·7% soln., 100 ml for 100 g of SiO_2). There is no interference from NO_2, Cl, ClO_2 or acetic acid; the outlet gas can be passed through a thymolphthalein tube for the determination of CO_2. K. Saito

1702. **The rapid analysis of sulphuric acid - nitric acid mixtures with a view to control of manufacturing processes.** O. Frehden and G. Ionescu. *Rev. Chim., Bucharest*, 1958, **9** (6), 311–313.—The method is based on the reaction of dithizone with the excess of lead nitrate soln. over that required to precipitate sulphate ions. Interfering heavy metals are first removed with Na_2CO_3 and alumina; if chloride is present, it is removed by $AgNO_3$ before the heavy metals, so that excess of $AgNO_3$ is removed with them. H. Sher

1703. **Heterometric titration of small amounts of sulphates.** C. J. van Nieuwenburg and B. F. Engelbert van Bevervoorde (Dept. of Micro-analysis, Tech. Univ., Delft, The Netherlands). *Anal. Chim. Acta*, 1958, **19** (1), 32–38.—The point of optimum turbidity in the quant. pptn. of $BaSO_4$ from K_2SO_4 (2 to 20 mg of SO_4^{2-}) by $BaNO_3$ was measured with a photo-electric cell connected to a galvanometer. The test beaker, which was shielded from stray light, was fitted with a magnetic stirring device. A minimum of 50% by vol. of alcohol in the liquid after titration was necessary for complete pptn. Of the alcohols tried, the sharpest end-point and most accurate result was given by a mixture of ethanol, *n*-butanol and water (2:1:1). The interference of alkali salts is not proportional to the concn. present. Interference by Ca, Al and Fe can be avoided by prior pptn. F. J. M.

1704. **The purification of barium sulphate by re-precipitation.** A. I. Busev and L. V. Kiseleva (Anal. Chem. Dept., Moscow Univ.). *Vestn. Moskov. Univ.*, 1957, (5), 227–228.—Filtered and washed $BaSO_4$ (≃ 0·11 g), obtained by pptn. of a soln. of $BaCl_2$ with H_2SO_4, was dissolved in a 7% soln. of EDTA (disodium salt) containing NH_3, the soln. was diluted to 300 ml, treated with 0·5 ml of dil. H_2SO_4 (1:2), boiled, and then made acid to methyl red indicator with HCl or H_2SO_4 soln. The cryst. ppt. was collected, washed and ignited. The error is much less than with other variants of the method. G. S. Smith

1705. **Separation of sulphate, sulphite, thiosulphate and sulphide ions with anion-exchange resins.** Akira Iguchi (Univ., Tokyo, Japan). *Bull. Chem. Soc. Japan*, 1958, **31** (5), 600–605 (in English).—The distribution coeff. (K_d) of S^{2-}, SO_3^{2-}, SO_4^{2-} and $S_2O_3^{2-}$ between an anion-exchange resin and aq. soln. (pH 7 to 11) containing NO_3^- are listed. The effect of organic solvents on K_d is also reported. When in admixture, these ions can be separated successively by elution from a column of a strongly basic resin (Diaion SA 100) (17 cm × 0·7 cm, nitrate form). The eluents are $NaNO_3$ soln. (0·1 mole per litre) (**I**); $NaNO_3$ soln. (1 mole per litre) (**II**); $NaNO_3$ soln. (0·1 mole per litre, adjusted to pH 9·7 with aq. NH_3) (**III**); and NH_4NO_3 soln. (0·1 mole per litre, adjusted to pH 9·7, plus 30% of acetone) (**IV**). For a mixture of SO_3^{2-}, SO_4^{2-} and $S_2O_3^{2-}$ the column is eluted first with **III** to remove SO_3^{2-} and SO_4^{2-} in that order, and then with **II** to remove $S_2O_3^{2-}$. The flow rate should be 0·3 ml per min. When S^{2-} are also present, the column is eluted first with **IV** to remove SO_3^{2-} and S^{2-} in that order, then with **I** to remove SO_4^{2-}, and finally with **II** to remove $S_2O_3^{2-}$. The flow rate should be 0·1 ml per min. Provided that means are taken to avoid loss of H_2S from the eluate containing S^{2-}, the recovery is > 97% for each ion.

W. J. Baker

1706. **Separation of polythionates with anion-exchange resins.** Akira Iguchi (Univ., Tokyo, Japan). *Bull. Chem. Soc. Japan*, 1958, **31** (5), 597–600 (in English).—The distribution coeff. of polythionates between an anion-exchange resin and HCl soln. increases from $S_2O_6^{2-}$ to $S_5O_6^{2-}$. A mixture of these can be separated by adsorption on a column (11 cm × 1 cm, Dowex 1-X2, Cl^- form) followed by elution of $S_2O_6^{2-}$ with N HCl, of $S_3O_6^{2-}$ with 3 N HCl, of $S_4O_6^{2-}$ with 6 N HCl and, finally, of $S_5O_6^{2-}$ with 9 N HCl. The concn. of $S_2O_6^{2-}$ in the eluate can then be determined gravimetrically, and that of each of the other three polythionates by titration with KIO_3 soln. Other oxy-acids (SO_3^{2-}, SO_3^{2-} or $S_2O_3^{2-}$) are eluted with the N HCl before $S_2O_6^{2-}$ appear in the eluate. A procedure for the analysis of 1 ml of Wackenroder's liquid by this method is given. W. J. Baker

1707. **Influence of heavy metals on the polarographic waves of selenium and tellurium.** A. I. Zelyanskaya, I. E. Bȳkov and L. S. Gorshkova. *Trudȳ Inst. Metallurg. Ural'sk. Fil. Akad. Nauk SSSR*, 1957, (1), 161–169; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,579.—The influence of Cu, Pb, Zn and Fe on the polarographic waves of Te and Se in a medium of aq. NH_3 and NH_4Cl is studied. If the concn. of Cu is greater than that of Te, the Cu must be removed. Zinc has no influence on the wave due to Te, but Te decreases the wave due to Zn. In the polarography of Te in the presence of Pb it is necessary to add 0·04 mole of EDTA (disodium salt) per litre and 0·1% gelatin soln. to the basal electrolyte. The effect due to Fe is removed by complexing the Fe with tartrate and increasing the concn. of gelatin. The presence of Cu, Pb and Fe^{3+} causes interference in the determination of Se; in the presence of EDTA (disodium salt), Zn, Ni and Co^{2+} do not interfere; Te interferes only when its concn. is greater than that of Se.

C. D. Kopkin

1708. **Polarographic determination of quadrivalent selenium and tellurium when present together.** A. I. Zelyanskaya, I. E. Bȳkov and L. S. Gorshkova. *Trudȳ Inst. Metallurg. Ural'sk. Fil. Akad. Nauk SSSR*, 1957, (1), 155–160; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,580.—It is established that with concn. of Se 0·05 to 2 mM and concn. of Te not greater than this, the most suitable basal electrolyte has the composition NH_4Cl 0·75, aq. NH_3 0·25 and Na_2SO_3 0·1 mole per litre, with 0·002% of gelatin. To analyse a washed residue of elementary Se and Te, obtained by any method, add 5 ml of a freshly prepared soln. containing up to 25 mg of $KClO_3$ in HCl (1:1), mix and warm gently. Neutralise the resulting soln. with aq. NH_3 to methyl orange, cool, dilute to 50 ml with the basal soln. and polarograph. Nitrates and heavy metals interfere. The results obtained agree satisfactorily with those of chemical methods.

C. D. Kopkin

1709. **Separation of selenium and tellurium by the use of a cationite.** I. E. Bȳkov and L. S. Gorshkova. *Trudȳ Inst. Metallurg. Ural'sk. Fil. Akad. Nauk SSSR*, 1957, (1), 151–154; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,497.—The possible separation of Se and Te by ion exchange on the cationite Espatit KU-1 (H form) was studied. The cationite grains were charged into a tube of height 20 cm, diam. 2·5 cm, washed with 5% HCl and then with water till neutral. The content of Se and Te in the filtrate was determined polarographically in a basal soln. 0·5 M in NH_4Cl, 0·5 M in NH_3, 0·1 M in Na_2SO_3 and containing 0·002% of gelatin. Experiments with the individual soln. showed that both in acid and alkaline media Se passes completely into the eluate, while at pH 2·7 to 8·5 Te is completely adsorbed on the cationite. Aq. NH_3 (1:3 or 1:2) was used to elute Te. Experiments with soln. containing both elements showed that at pH 1·4 Se passes completely into the eluate, while Te is adsorbed on the cationite and may be eluted with aq. NH_3 (150 ml). The cationite is regenerated by washing with water, with 5% HCl, and again with water till neutral to methyl orange. At pH 1·5 to 3·7, Cu, Fe, Pb and Zn are completely adsorbed by this cationite; the separation of Se from Cu, Fe and Zn may thus be effected. From soln. containing Te together with Cu, Fe, Zn or Pb, the Te is adsorbed by the cationite, but is eluted with aq. NH_3 to the extent of only 70 to 85%. It is established that in weakly acid soln. containing EDTA (disodium salt), only Te and Pb are adsorbed, while in ammoniacal soln. containing K Na tartrate, only Cu and Zn are adsorbed.

C. D. Kopkin

1710. **Separation of tellurium(IV) from tellurium(VI) by solvent extraction with tributyl phosphate.** Mariko Inarida (Chem. Dept., Fac. of Sci., Tokyo Univ., Hongo, Tokyo). *Japan Analyst*, 1958, **7** (7), 449–451.—Whilst Te^{IV} is quant. extracted with pure tributyl phosphate (**I**) from 2 to 10 N HCl, Te^{VI} is not extracted from 0·5 to 10 N HCl, and their separation is satisfactorily effected in 2 to 10 N HCl. This method is superior to the use of **I** in kerosine (20%) (*cf. Anal. Abstr.*, 1959, **6**, 1284).

K. Saito

1711. **Rapid photometric determination of chromium in copper - chromium alloys.** H. Wiedmann. *Z. Metallk.*, 1958, **49** (5), 248–249.—Shavings (0·5 g) are dissolved in HNO_3 (1:1) (10 ml) then 15 ml of an acid mixture [H_2SO_4 (1:1) (660 ml) and H_3PO_4 (200 ml)] is added and the mixture is heated until fumes are evolved and the soln. becomes clear. After a few seconds 20 ml of $HClO_4$ (sp. gr.

1·7) is added and heating continued for 75 seconds. The soln. is diluted to 200 ml with H_2O and the extinction is measured with the use of filter S47E in a 5-cm cell against water. The Cr content is calculated from a standard curve. One analysis requires 10 to 15 min. For concn. of Cr of 0·73 and 1·43%, results are accurate to within ± 0·02%. N. E.

1712. **Notes on the analysis of chromite.** I. Sarudi (v. Stetina) (Chem. Untersuchungsamt. der Stadt Szeged, Hungary). *Z. anal. Chem.*, 1958, **163** (1), 34–37.—A method is given for the separation of Cr from Al in the aq. soln. obtained after fusing chromite with NaOH and KNO_3. The Cr is oxidised with conc. HNO_3 and solid $KClO_3$ and then separated from Al by pptn. as lead chromate. On a sample containing 300 mg of Cr as Cr_2O_3 the accuracy of the method is approx. 0·3%. *Procedure*—Acidify the aq. extract from the fusion with HNO_3 and evaporate almost to dryness. Add conc. HNO_3 (15 ml) and solid $KClO_3$ (0·4 to 0·5 g) and heat on a water bath. Add more solid $KClO_3$ at intervals over a period of 1 to 1·5 hr. until a total of 1 g has been added. Evaporate almost to dryness, add water (30 to 40 ml), filter, wash, and ignite the SiO_2. Add conc. aq. NH_3 to the filtrate until just alkaline. Filter off the ppt., dissolve it in conc. HNO_3 and oxidise with $KClO_3$ as described above. Recombine the oxidised soln. with the main filtrate and make weakly alkaline with conc. aq. NH_3. Add glacial acetic acid (3 ml) to dissolve the ppt. of $Al(OH)_3$. Heat to boiling and precipitate $PbCrO_4$ with Pb acetate soln. (7·3 g in 100 ml). Allow to stand for 1 to 1·5 hr. at room temp., filter, wash the ppt. with cold water and dry at 110° to 120°. Weigh as $PbCrO_4$ and calculate as Cr_2O_3, with an empirical factor of 0·2328. W. T. Carter

1713. **New reagents for tungsten. 4:4′-Bis-dimethylaminotriphenylmethane [leuco-base of malachite green].** J. Zato (Univ. Laboral José Antonio Girón, Gijón). *Inf. Quim. Anal.*, 1958, **12** (3), 68–75.—Dissolve 4:4′-bisdimethylaminotriphenylmethane (20 g) in dil. HCl (200 ml of H_2O, 15 to 20 ml of conc. HCl), filter and dilute to 1 litre. Add 10 ml of this soln. for each 250 mg of WO_3^- present in the hot acid soln. (up to 3 *N*) of the sample. Filter off the ppt. on a sintered crucible, wash with a dil. soln. of reagent and ignite at 400° to 600°. The ppt. has the composition $5WO_3.C_{23}H_{24}N_2$. Results are in good agreement with those obtained by pptn. with cinchonine. Phosphate, MoO_4^- and large amounts of SiO_2 (> 10 mg) interfere. G. H. Foxley

1714. **Gravimetric determination of tungsten in the presence of molybdenum.** A. Claeys (Univ. of Ghent, Belgium). *Anal. Chim. Acta*, 1958, **19** (2), 114–118.—Co-precipitation of Mo occurs when W is pptd. with cinchonine or 5:6-benzoquinoline (**I**). Treatment of the cinchonine ppt. with hot conc. H_2SO_4 removes some of the Mo, but not all. A process of double pptn., the ppt. being extracted with hot dil. HNO_3, reduces the error to 0·6% when the Mo:W ratio is 1:2, and to ≃ 1% when the ratio is 1:1. *Procedure*—To 25 ml of soln. containing ≃ 170 mg of WO_3 add 10 ml of conc. HCl, a few drops of conc. HNO_3, 2 ml of 30% H_2O_2, and filter pulp. Heat to boiling, add 3 ml of **I** soln. (2 g in 10 ml of 14 *N* HNO_3 diluted to 100 ml) and digest on a steam bath. Decant the clear liquid through a filter-paper and add the paper to the ppt. Heat the ppt. with water (15 ml), 14 *N* HNO_3 (6 ml), 30% H_2O_2 (2 ml) and **I** soln. (3 ml) and filter. Dissolve the ppt. in 5% NaOH soln. (30 ml) on the steam bath, add warm water (30 ml), filter and wash the filter with 1% NaOH soln. Evaporate the filtrate slowly to 50 ml and add 3 ml of 30% H_2O_2. Heat to boiling, add conc. HCl (20 ml), 14 *N* HNO_3 (1 ml) and **I** soln. (8 ml) and extract the ppt. as in the first pptn. Finally wash the ppt. on the filter with **I** soln. (5 ml) diluted with 0·2 *N* HNO_3 (95 ml), dry, and ignite at 850°. H. N. S.

1715. **Co-precipitation of tungsten for its determination in products containing molybdenum.** A. N. Zelikman and N. N. Gorovits (M. I. Kalinin Moscow Inst. of Non-Ferrous Metals and Gold). *Zavod. Lab.*, 1958, **24** (8), 940–941.—The soln. of the material in HCl was mixed with $FeCl_3$ and the W was co-pptd. with $Fe(OH)_3$ on the addition of aq. NH_3. The coagulated ppt. was dissolved in HCl, Fe was removed by means of NaOH, and the W was determined colorimetrically by the thiocyanate method. By the use of ^{185}W with a preparation containing 100 parts of Mo to 1 of W only 70 to 79% of the W was recovered. The problem of determining W in the presence of Mo is still considered unsolved. G. S. Smith

1716. **Spectrographic determination of impurities in tungstate.** Hiroshi Hamaguchi, Rokuro Kuroda and Ryokichi Negishi (Chem. Dept., Fac. of Sci., Tokyo Univ. of Education, Koishikawa, Tokyo). *Japan Analyst*, 1958, **7** (7), 415–419.—The addition of graphite (Lounamaa, *Spectrochim. Acta*, 1951, **4**, 400) not only suppresses the appearance of numerous emission lines of W, but also that of impurities to be detected. It was found that the use of an inorganic salt (*e.g.*, $BaCl_2$, NaCl or GeO_2) in addition to graphite promotes the volatilisation of metallic impurities in ammonium tungstate (**I**), while the lines of W remain suppressed. The addition to **I** of 5 times the wt. of graphite and of 5% by wt. of $BaCl_2$ are the best conditions for the detection of Si, Ni, V, Pb and Mg in **I** (intermittent arc, 220 V, 7 amp.; electrode gap, 3 mm; electrode, graphite, 0·25 in.). The suppressive action of graphite appears to be due to the formation of metallic tungsten and tungsten carbide. K. Saito

1717. **Chromatographic separation of tungsten and molybdenum as sulphur compounds on aluminium oxide.** T. A. Davrisheva. *Soobshch. Akad. Nauk GruzSSR*, 1957, **19** (4), 421–428; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,494.—The described method is based on the quant. adsorption of the complex sulphur compound of W on partially dehydrated aluminium hydroxide, the molybdenum complex not being adsorbed. To prepare the adsorbent, add 10% H_2SO_4 to a soln. of sodium aluminate, filter off the ppt. formed, wash it with water till almost completely free of SO_4^{2-}, dry it at 70°, grind (120 mesh) and dehydrate at 800° for several minutes. The residual moisture content (20%) is desirable and ensures poor adsorption of Mo but practically complete adsorption of W. The optimum values of other factors are—the pH of the polysulphide soln. of the sulphur derivatives of Mo and W should be ≃ 11, the composition of the sodium polysulphide used should correspond to the formula Na_2S_2, and 100 ml of the polysulphide soln. should contain ⩾ 50 mg of W and 10 mg of Mo. To determine the amount of adsorbed W, wash the column with bromine water to oxidise sulphides,

with water and then with hot aq. NH_3, and determine W in the filtrate. Precipitate Mo as the sulphide MoS_3 by acidification of the polysulphide filtrate. Interference to the chromatography is caused by elements that form poorly soluble sulphides or hydroxides in the polysulphide soln., *e.g.*, Ba and Ca, or that form insol. salts with the sulphur derivatives of W and Mo. The method has been satisfactorily used for the separation of W and Mo in the analysis of scheelite. C. D. KOPKIN

1718. **Use of ascorbic acid for determining molybdenum by the thiocyanate method.** A. I. Lazarev and V. I. Lazareva (Kuibishev Industrial Inst.). *Zavod. Lab.*, 1958, **24** (7), 798–800.—In the colorimetric thiocyanate method for determining Mo, the optimum conditions when ascorbic acid is used as reducing agent are—H_2SO_4 1·4 *M*, KSCN 0·6 *M*, ascorbic acid 0·05 *M* (it is immaterial whether this is added before or after the KSCN), time for colour development $\lessdot$ 10 min., wavelength for extinction measurements $\simeq$ 440 mμ. The presence of Cl^- and of $\gtrsim$ 0·15 *M* NO_3^- does not cause interference, but in the analysis of steel the reduction of Fe^{3+} is greatly retarded in the presence of both Cl^- and SCN^- and it is necessary to remove Cl^-. To determine Mo in chrome steel, the sample (0·1 g) is heated with 10 ml of 20% H_2SO_4 followed by oxidation with 1 ml of HNO_3, and the soln. is evaporated to fuming with subsequent dissolution of the salts by heating with 50 ml of water and then dilution to 250 ml. With tungsten steel, the sample is dissolved in 10 ml of 20% HCl, followed by addition of HNO_3 and evaporation to dryness and dissolution in 10 ml of 50% citric acid soln. and 40 ml of water. With niobium steel, after treatment as for tungsten steel the insoluble hydrated Nb_2O_5 is filtered off and washed with hot 5% HCl soln. The soln. is diluted to 250 ml. An aliquot (the amount taken should give an optical density of $\simeq$ 0·43) is mixed with 4 ml of dil. H_2SO_4 (1:1) and water to give a volume of 15 ml, then cooled and mixed with 4 ml of 4 *M* KSCN and 5 ml of ascorbic acid reagent soln. (prepared by dissolving 10 g of ascorbic acid and 2 ml of 0·1 *N* EDTA in 90 ml of water and adding 10 drops of formic acid) followed by dilution to 25 ml. After 20 min. the extinction of the soln. in a 30-mm cell is measured with a 440-mμ filter. Comparison is made with a soln. containing all the reagents except KSCN.
G. S. SMITH

1719. **The use of specific metal indicators in precipitation titrations. I. End-point determination in the titration of molybdate with lead solutions.** R. Püschel, E. Lassner and R. Scharf (Tech. Hochsch. Graz, Austria). *Z. anal. Chem.*, 1958, **163** (2), 104–110.—The use of specific metal indicators for lead in the pptn. titration of molybdate with lead solutions has been studied. The indicators chosen were (*i*) PAN - Cu - EDTA and (*ii*) PAR [4-(2-pyridylazo)resorcinol]. Xylenol orange was tested but gave a colour change with molybdate and was therefore excluded. (*i*) The molybdate solution is diluted to 50 to 100 ml, brought to pH 6 and heated to boiling. After the addition of 1 drop of 0·01 *M* Cu - EDTA and 3 to 5 drops of PAN indicator solution (0·1% in alcohol), titration is carried out with lead nitrate solution at a rate of 1 drop per 2 to 3 sec. until a colour change from yellow or orange to raspberry red is observed. The temperature of the solution should not drop below 80° during titration. If more than 10 mg of Mo is present, the solution is titrated without indicator until precipitation begins, when indicator is added and titration completed as above. (*ii*) After addition of 3 to 5 drops of PAR solution (0·1% in water) titration with lead nitrate is carried out at a rate of 1 to 2 drops per sec. with stirring until the colour changes from yellow - green to red. Absolute errors are ± 0·2 mg of Mo with 0·05 *M* lead solution and 0·02 mg of Mo with 0·005 *M* lead solution. Interference is produced by cations which give a colour with the indicator, *e.g.*, Fe, Ni, Cu, Co and U, but in most cases these can be removed quite simply. Aluminium is precipitated at the pH of the titration but can be removed by filtration. Dichromate, tungstate and vanadate interfere, as do anions which increase the solubility product of lead molybdate, *e.g.*, acetate, tartrate and citrate.
S. M. MARSH

1720. **The quantitative determination of uranium by separation with ion exchange and Complexone.** M. Fodor (Central Phys. Res. Inst. of the Hungarian Acad. of Sci.). *Magyar Kém. Foly.*, 1958, **64** (7–8), 229–230.—To the soln. of the U-containing ore, add $\simeq$ 50% excess of EDTA (disodium salt) and adjust the pH to 7 with carbonate-free aq. NH_3. At this pH, no ammonium uranate separates. To prevent the displacement of the uranium from the column by the conc. soln., dilute it to 50 or 100 ml, according to its salt concn. Pass the soln. through a column (10 cm × 1 cm) of acid Amberlite IRC-50 resin at a rate of 3 ml per min. Convert the resin into its acid form with 3 *M* H_2SO_4 (50 ml) and wash it with water (100 ml) until acid-free. Pass the eluate through a 10 cm × 1 cm reductor, containing metallic bismuth or cadmium of 0·6 to 1 mm diameter. This reduces U^{VI} to U^{IV}; oxidise any U^{III} to U^{IV} by blowing air through the soln. for 10 min. Titrate the U^{IV} in the soln. with 0·005 *N* NH_4VO_3, with phenylanthranilic acid as indicator (0·2% phenylanthranilic acid dissolved in 0·2% Na_2CO_3 soln.), or with 0·005 *N* Ce^{IV}, with ferroin as indicator. The recovery of U is quant. The method is suitable also for the assay of ores.
A. G. PETO

1721. **Separation of uranium, vanadium and iron by paper chromatography.** Yu. V. Morachevskiĭ, M. N. Gordeeva and T. E. Kruglova (Leningrad State Univ.). *Zavod. Lab.*, 1958, **24** (7), 790–793.—The use of circular paper chromatography with a mixture of glacial acetic acid and HNO_3 (95:5) as the moving solvent is suitable for quantitative separation and can be followed by the determination of U (5 to 500 μg), Fe (50 to 750 μg) and V ($\lessdot$ 2·5 μg).
G. S. SMITH

1722. **Use of the radiometric method for determining uranium, thorium and radium in ores.** E. I. Zheleznova and D. V. Tokareva (All-Union Inst. of Mineral Raw Materials). *Zavod. Lab.*, 1958, **24** (8), 959–963.—Measurements of total γ-ray intensity, the harder γ-ray intensity ($>$ 1·8 MeV for Th), intensity of accumulated Ra emanation, and aluminium-filtered β-ray intensity (for U) are made, and the contents of U, Th and Ra are calculated.
G. S. SMITH

1723. **Phthalocyanines as oxidation - reduction indicators. II. Use of copper phthalocyaninetetrasulphonic acid (Cu - PTS). Cerimetric determination of uranium(IV), molybdenum(V) and arsenic(III).** T. P. Sastri and G. Gopala Rao (Dept. of Chem., Andhra Univ., Waltair, India).

Z. anal. Chem., 1958, **163** (1), 1–6 (in English).—With copper phthalocyaninetetrasulphonic acid as indicator, U^{IV} is titrated with ceric sulphate in 2 *N* H_2SO_4 containing H_3PO_4 (5%, v/v) and Mo^V in 0·5 to 3 *N* H_2SO_4 containing H_3PO_4 (3%, v/v). The end-point change is from blue via a transient pink to colourless; H_3PO_4 is added to prevent a premature end-point. Tervalent As is titrated in the cold in *M* H_2SO_4 containing several drops of 0·01 *M* OsO_4 as catalyst. The end-points are sharp and although the indicator is not reversible it has a small indicator correction and is stable in solution.

W. T. CARTER

1724. **Colorimetric determination of plutonium in microgram quantities.** T. V. Healy and P. E. Brown (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report C/R 1287, 1957, 16 pp.—The procedure involves the addition of up to 150 μg of Pu^{IV} to 2·5 ml of 0·1% 1-(*o*-arsenophenylazo)-2-naphthol-3:6-disulphonic acid (thoron) and dilution to 25 ml; the soln. should then be between 0·05 and 0·25 *N* with respect to HCl or HNO_3. The optical density at 540 mμ is then measured, within 30 min. normally, but within 5 min. if HNO_2 is present. The concn. of Pu is obtained by reference to a standard curve. Tervalent Pu may be converted into Pu^{IV} by $NaNO_2$ and Pu^{VI} into Pu^{III} by hydroxylamine hydrochloride and thence to Pu^{IV}, before determination. The accuracy is ± 1% at the 100-μg level. Methods are discussed for the elimination of interferences due to UO_2^{2+}, Fe^{3+}, F^-, oxalate and several other ions.

G. J. HUNTER

1725. **Some experiments on the polarography of plutonium(VI) in complexing media.** G. P. Cook, J. K. Foreman and E. F. Kemp (U.K. Atomic Energy Authority, Windscale Works, Cumberland, England). *Anal. Chim. Acta*, 1958, **19** (2), 174–179.—In order to bring the polarographic reduction of Pu into the working range of the dropping mercury electrode it is necessary to complex the oxidised form more strongly than the reduced form. To this purpose the reduction of Pu^{6+} (1 mg per 1 ml) is conducted in *N* NaOH, 0·1 *M* K Na tartrate, *M* acetic acid - Na acetate (pH 4·0), and 0·5 *M* Na oxalate - oxalic acid (pH 4·0), respectively. The $E_{\frac{1}{4}}$ obtained in each electrolyte is recorded. Only the wave obtained in oxalate soln. is reversible and this corresponds to the reduction of Pu^{6+} to Pu^{5+}. The non-linear dependence of diffusion current upon Pu^{6+} concn. for this reduction is consistent with the induced disproportionation of Pu^{5+} in oxalate soln.

H. N. S.

1726. **Isotopic analysis of plutonium and uranium by mass spectrometer.** A. H. Turnbull and D. F. Dance (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report C/R 2776, 1958, 6 pp.—Experience with the Metropolitan Vickers Type MS 5 mass spectrometer is described. Sample sizes are 10^{-8} g for Pu and 10^{-6} g for U, and a coefficient of variation of ± 10% is achieved for an isotopic abundance of 0·01%. Other aspects considered are checking of resolution and speed of operation. G. J. HUNTER

1727. **Determination of small amounts of hydrogen fluoride by means of titanium - chromotropic acid reagent.** M. D. Skanavi. *Zavod. Lab.*, 1958, **24** (6), 683.—The reagent is prepared by dissolving the washed ppt. of $Ti(OH)_4$ (obtained by the addition of aq. NH_3 to a soln. of 50 mg of $TiOSO_4$ in 20 ml of conc. H_2SO_4) in 4 ml of 3 *N* H_2SO_4, diluting to 300 ml and mixing 3 vol. of this soln. with 3 vol. of a 0·05% freshly prepared aq. soln. of chromotropic acid and 5 vol. of 0·1 *N* Na acetate. To determine F^- (1 to 10 μg per ml), the sample (1 or 2 ml) in a flat-bottomed colorimeter tube is mixed with 1 ml of the reagent. The colour can be compared at once with a standard, but it is stable after long standing. In the absence of F^-, the colour is reddish brown. The presence of F^- causes bleaching and the colour tends to yellow. The method is suitable in the presence of SO_4^{2-}, which interfere in the methods based on the use of coloured complexes of Zr, Th and Fe. It is applied to the determination of HF (0·3 to 17 μg per litre) in air. G. S. SMITH

1728. **Analytical determination of chlorites and chlorine dioxide.** Sh. S. Shchegol'. *Trudy Khim. i Khim. Tekhnol.*, 1958, (1), 148–156; *Ref. Zhur., Khim.*, 1958, (24), Abstr. No. 81,353.—Spectrophotometric and polarographic methods are evolved for the determination of $NaClO_2$ and ClO_2 in aq. soln. when present together. (*i*) Measure the extinction of a soln. 1 to 2 × 10^{-3} *M* with respect to $NaClO_2$ and ClO_2, first at 400 mμ (to determine ClO_2), and then at 260 mμ (to determine total ClO_2 + $NaClO_2$); the relative error of the method is ± 8 to 9%. (*ii*) Place 0·1 *M* K_2SO_4 in a normal polarographic cell containing buffer soln. (750 g of $NaH_2PO_4.H_2O$ per litre, pH 7), pass in H or N, introduce the soln. to be analysed in a glass ampoule, and polarograph from 0 to 0·5 V (S.C.E.). Determine ClO_2 in one portion of the soln. in the presence of $NaClO_2$ and in another portion convert ClO_2 into $NaClO_2$ by making the soln. alkaline to pH 11, and find total $NaClO_2$ + ClO_2 by polarography in a basal soln. of 0·5 *M* KCl - 0·001 *M* $LaCl_3$. The method cannot be used if the soln. also contains Cl or HClO or its salts. The relative error of the method is ± 8%. It is established that the amperometric method is more accurate than spectrophotometric, polarographic or volumetric methods, particularly in the determination of small concn.

C. D. KOPKIN

1729. **New variant of the determination of active chlorine in chlorinated lime by the method of electrometric titration.** G. V. Zavarov (Chernorechensk Chem. Works). *Zavod. Lab.*, 1958, **24** (6), 681–682.—The electrodes are platinised platinum (anode) and tungsten (cathode), the titrant is *N* Na_2HAsO_3 containing $NaHCO_3$ (20 g per litre), and the titration is carried out by adding rapidly 5 to 10 ml of the titrant to the greater part of an aliquot (25 ml) of a soln. of 7·1 g of the sample of chlorinated lime in 500 ml at 2·5 to 3 V, then adding the reserved portion of the aliquot and completing the titration slowly at 0·7 to 1 V. G. S. SMITH

1730. **Determination of iodate and periodate ions in the presence of each other.** L. Szekeres, M. Rády and E. Kardos (Chem. Inst., Agric. Univ., Budapest, Hungary). *Z. anal. Chem.*, 1958, **162** (6), 430–431.—In a previously described method for the determination of IO_3^- and IO_4^- in admixture (*cf. Anal. Abstr.*, 1958, **5**, 4111), the Br released by the reduction of IO_4^- and IO_3^- with KBr and HCl was removed by alkali and ethanol. Urea is now suggested as a substitute for ethanol, any excess not interfering with the subsequent iodimetric determination of the IO_3^-. J. H. WATON

1731. **Direct oxidative determination of manganese.** J. Doležal and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chemie, Prague*, 1958, **10** (5), 369–375.—A critical review. A modification of the Lingane and Karplus method (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 191) (potentiometric titration with $KMnO_4$ in pyrophosphate medium of pH 7) has been found the most suitable method. *Procedure for manganese concentrates*—Heat the sample (0·5 g), containing 5 to 20% of Mn, with conc. HNO_3 (30 ml) and conc. HCl (10 ml) and evaporate with conc. H_2SO_4 (5 ml). Transfer the residue with H_2O into a 100-ml flask, cool and dilute to vol. with H_2O. To 25 ml of the soln. (the remaining undissolved residue causes no interference) add 3 to 5 drops of H_2O_2 (30%), remove the excess by heating, saturate the soln. with sodium pyrophosphate and adjust the pH to $\simeq$ 7 with HCl or KOH. Titrate potentiometrically with 0·025 *M* $KMnO_4$, with the use of platinum - tungsten electrodes. J. ZÝKA

1732. **Determination of rhenium in products containing molybdenum.** B. N. Ranskiĭ (Balkhash Copper Melting Works). *Zavod. Lab.*, 1958, **24** (7), 803–804.—If the content of Mo does not exceed that of Re, the sample (1 to 2 g) in a crucible is mixed with 3 g of hydrated CaO and moistened with saturated $Ca(NO_3)_2$ soln., then covered with a layer of hydrated CaO, and ignited at 600° for 2 to 3 hr., the mixture being stirred half-way through. The cooled mass is heated with 150 ml of water and 25 ml of saturated bromine water for 30 to 60 min., and the soln. after filtration is treated with H_2SO_4 to give a pH of 4·5 to 5·0. The colorimetric thiocyanate method is applied. With higher contents of Mo, 100 ml of the soln. (pH 4·5 to 5·0) is passed through a column of Sulphocarbon and the first 50 ml is rejected. G. S. SMITH

1733. **Selective separation of iron by solvent extraction.** H. Specker (Inst. für Spektrochemie, Dortmund-Aplerbeck, Germany). *Arch. Eisenhüttenw.*, 1958, **29** (8), 467–470.—High selectivity and good reproducibility are obtained by solvent extraction with *iso*butyl methyl ketone from HCl or from LiCl soln. The method allows for quant. separation from Fe of 25 elements and enrichment of traces of heavy metals in an additional extraction system. H. SAWISTOWSKI

1734. **Determination of iron in blast-furnace iron ores.** J. Michal and J. Žalud (Res. Inst. Ores, Prague). *Hutn. Listy*, 1958, **13** (7), 639–641.—*Procedure*—Dissolve the finely powdered sample (0·2 to 0·3 g) by heating with a mixture of $HClO_4$ and H_3PO_4 (1 : 1) on a sand bath, cool, dilute with 100 ml of 20% HCl, boil, cool and pass through a column of silver reductor. Wash the reductor with 200 ml of 20% HCl and titrate potentiometrically with 0·1 *N* $K_2Cr_2O_7$. J. ZÝKA

1735. **Use of iodine chloride in analytical chemistry. IX. Determination of iron(II), titanium(III) and EDTA (disodium salt).** J. Čihalík (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (6), 1075–1082.—The slow direct oxidation of Fe^{2+} with ICl can be accelerated in the presence of complex-forming compounds [$Na_4P_2O_7$, NH_4F and EDTA (disodium salt) (**I**)]. If **I** is added to Fe^{2+}, the direct potentiometric titration of Fe^{2+} with ICl in an inert atmosphere can be carried out. Two potential changes ($I^+ \rightarrow I^-$ and $I^- \rightarrow I$) can be observed. The reaction can be used also for the oxidative determination of **I**. Tervalent Ti can be titrated directly in an inert atmosphere; the course of titration is similar to that of Fe^{2+}. In the presence of **I** and NH_4F a mixture of Ti^{3+} and Fe^{2+} can be determined; the first potential change corresponds to $Ti^{III} \rightarrow Ti^{IV}$, the second indicates the oxidation of $Fe^{II} \rightarrow Fe^{III}$. The error on a semi-micro scale did not exceed $\pm$ 0·5%. J. ZÝKA

1736. **Detection of ferricyanides in the presence of certain oxidants.** A. P. Kreshkov, S. S. Vil'borg and V. A. Drozdov. *Nauch. Dokl. Vyssh. Shkoly Khim. i Khim. Tekhnol.*, 1958, (2), 314–316; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,590.—New colour reactions are described for the detection of ferricyanides (**I**) by the use of certain amines, based on oxidation of the amines to form dyes. To detect **I**, to 1 ml of soln. add 3 drops of a 5% soln. of the amine (or mixture of amines) in acetic acid, and 1 drop of a saturated soln. of $ZnSO_4$ (to raise the oxidation - reduction potential of the ferri - ferrocyanide system). The most sensitive amines for this method are 4 : 4′-diamino-3 : 3′-dimethyldiphenylmethane and its NN′-diethyl, -dipropyl and -dibutyl derivatives; for the first three reagents the sensitivity is 4·8 μ_K of $K_3Fe(CN)_6$ per ml, and for the last 7·4 μg per ml. No interference is caused by nitrates, chlorates, bromates, iodates, perchlorates, arsenates or periodates; nitrites, persulphates and chromates interfere. C. D. KOPKIN

1737. **Use of carbon electrodes for the spectrographic analysis of steel.** E. S. Kudelya (E. O. Paton Inst. of Electro-welding, Acad. Sci., UkrSSR). *Zavod. Lab.*, 1958, **24** (6), 752–753.—In sparking a sample of steel with a counter electrode of carbon or graphite, carbides form on the surface of the steel and affect the passage of alloying elements into the spark. The interference is particularly great in the determination of W. An electrode of copper is preferable. G. S. SMITH

1738. **Determination of non-metallic inclusions in iron and steel by the ethanolic iodine method.** Shizuya Maekawa and Miyoshige Ebihara (Muroran Works, The Japan Steel Works, Muroran, Hokkaido). *Japan Analyst*, 1957, **6** (11), 715–719.—Rooney's ethanolic iodine method for the separate determination of MnO and FeO [*J. Iron St. Inst.*, 1935, (1), 249] was simplified to minimise the time required. The use of a magnetic stirrer decreases the time taken for dissolution of the sample (1·5 hr. for turnings; 3 hr. for discs). Rapid filtration of a cold soln. does not vitiate the method. K. SAITO

1739. **A method of determining combined and free carbon by selective oxidation - reduction of iron carbides.** F. Marion and R. Faivre (École N.S. de la Métall. et de l'Ind. des Mines de Nancy). *Compt. Rend.*, 1958, **247** (2), 206–208.—The combined carbon is determined by selective oxidation - reduction of iron carbides in contact with an atmosphere of hydrogen saturated with water vapour. The hydrocarbons produced are then oxidised by CuO heated to 920° to 950°. The CO_2 formed is determined by passing it through a standardised soln. of $Ba(OH)_2$. The conditions of the reaction are described, and these depend largely on the degree of division of the test substance. K. R. C.

1740. **Determination of free carbon and combined carbon by selective oxidation - reduction. Application to mixtures of cementite, metallic iron and graphite and also to industrial cast iron.** F. Marion (Lab. de Phys. de l'État Metallique, Fac. des Sci., Nancy, France). *Bull. Soc. Chim. France*, 1958, (8–9), 1181–1187.—The action of H, either dry or with 10% or 45% of water vapour, is investigated on mixtures of cementite, graphite and iron. While the action of dry H is slow, in the presence of water vapour the curve of total C content against time for H plus H_2O shows a sharp inflection due to the rapid reaction of combined C and a slower attack on the uncombined C, possibly due to the formation of carbide in the furnace. The residue is Fe for H and H + 10% of H_2O, and oxides of Fe for H + 45% of H_2O. The most satisfactory temp. of reaction for the artificial mixtures is 500°. The uncombined C is determined by extrapolation of the second part of the curve back to the total C axis. In industrial cast irons the reaction is similar, but attack is more rapid with the 10% water mixture, and the optimum temp. for a nearly horizontal second part to the curve is 600° to 700°. Analyses by this and other methods are compared.
E. J. H. Birch

1741. **Determination of gases in steel by vacuum fusion - mass spectrometry.** J. F. Martin, J. E. Friedline, L. M. Melnick and G. E. Pellissier (U.S. Steel Corp., Monroeville, Pa.). *Trans. Met. Soc.*, 1958, AIME 212, 514–519.—A method has been developed for determining gases in steel in which the gases are extracted by vacuum fusion and analysed by mass spectrometry. This method is especially applicable for determining small amounts of O and N. The lower limit of detection for both O and N is 0·0001%, with an average deviation of about 0·0001% for O and 0·00008% for N. Results of hydrogen determinations by this method are in good agreement with those of determinations by vacuum tin-fusion. A complete analysis of all gases extracted from a steel sample can be accomplished in 40 min.
Nucl. Sci. Abstr.

1742. **Sampling and determination of hydrogen in steel under works conditions.** H. Zitter and H. Krainer (Forschungsanstalt, Gebr. Böhler A.-G., Kapfenberg, Austria). *Arch. Eisenhüttenw.*, 1958, **29** (7), 401–410.—The sample (5 g) is introduced magnetically into a quartz tube and heated by a movable furnace at 1000° for 20 min. The H evolved is pumped by a mercury diffusion pump into a collecting bulb with McLeod gauge, and measured. The apparatus is integral, with mercury cut-offs in place of taps and no fitted joints. Blank tests are usually negligible (≃ 0·003 ml per 100 g). The chief source of error is loss of H during sampling, due to the rapid fall of solubility with temp. Prompt cooling of the molten sample is essential to retard the escape of H by diffusion. A method of doing this (tentatively recommended by the Standardisation Committee of the Verein Deutscher Eisenhüttenleute) is to ladle the sample through a detachable funnel into a two-piece copper mould to form a cylinder 12 mm in diameter, which is dropped into water as soon as it reaches 700° to 800°, and is then further cooled (*e.g.*, by liquid air) to minimise diffusion of H during trimming, transport, etc. Polishing reduces the adsorption of air by the specimen, and the small amounts involved (≃ 0·2 ml per 100 g) are estimated by re-treating the out-gassed specimen after exposure to air. Data from a sample taken by dipping into the molten steel an evacuated glass tube ("pintube") drawn out at each end showed fair agreement with those from the mould samples, but the glass must have carefully adjusted softening- and melting-points, and it did not always part cleanly from the sample.
A. R. Pearson

1743. **Determination of boron in carbon and low-alloy steels.** B.I.S.R.A. Methods of Analysis Committee. *J. Iron St. Inst.*, 1958, **189** (3), 227–232.—The separation of boron from iron and interfering elements, and its absorptiometric determination with quinalizarin, curcumin and dianthimide were investigated. The recommended procedure is based on ion exchange and the use of dianthrimide, and is applicable to mild and low-alloy steels (< 2% of Cr, < 5% of V) for the determination of boron contents in the range 0·0005 to 0·02%.
J. W. O. Pyemont

1744. **Polarographic determination of lead in free-cutting steel without separation.** Kazuo Ota (Toyota Motor Co., Koromo, Aichi-ken). *Japan Analyst*, 1958, **7** (7), 429–432.—The polarographic determination of lead ($E_{\frac{1}{2}} = -0{\cdot}48$ V *vs.* the S.C.E.) in an HCl soln. is applied to the analysis of free-cutting steel. For the reduction of Fe, a mixture of hydrazine hydrochloride (**I**) and Na formate (**II**) gives best results. The presence of Mo affects the wave form. *Procedure*—Dissolve the sample (2·5 g) in HCl (1 + 1, 30 ml), heat with H_2O_2 (10%, 5 ml) and make up to 50 ml. To a 10-ml portion add aq. NH_3 soln. until a ppt. is formed, followed by HCl (1 + 1, 3 ml), **I** (0·2 g per ml, 10 ml), **II** (0·5 g per ml) and starch soln. (2%, 3 ml). Heat in a boiling-water bath for 5 min. and make up to 50 ml. Record the polarogram at − 0·3 to − 0·7 V.
K. Saito

1745. **Rapid spectrophotometric determination of phosphorus in carbon and alloy steels by the extraction method.** Mitsuru Ura (Ind. Res. Inst., Kanagawa Prefecture, Tomioka, Kanazawa-ku, Yokohama). *Japan Analyst*, 1958, **7** (7), 420–424.—The extraction of molybdophosphoric acid with *iso*butyl alcohol and $CHCl_3$ (*cf.* Kiba and Ura, *Anal. Abstr.*, 1956, **3**, 686) was applied to the determination of P (0·01 to 0·3%) in carbon and alloy steels. The extraction is almost complete without repetition by the use of 20 ml of the mixed solvent (2 + 3, by vol.). Silicon (0·5 to 3%), As (< 2%) and W (< 0·1%) do not interfere. Vanadate (< 5%) must be reduced to vanadyl with Fe^{2+}, and CrO_4^{2-} to Cr^{3+} with $NaNO_2$ before extraction. *Procedure*—Dissolve the sample (0·2 g) in HNO_3 (1 + 1, 7·5 ml) and oxidise the product with $KMnO_4$ soln. (2%) until MnO_2 is pptd. Add a little $(NH_4)_2SO_4.FeSO_4$, heat with water (50 ml), add ammonium molybdate soln. (10%, 3 ml), cool and shake with the solvent (20 ml) for 1·5 min. Measure the extinction of the org. layer at 372 mμ. Dissolve cast iron in HNO_3 (1 + 1, 7·5 ml) containing a few drops of HF and take an aliquot for the extraction of P. Dissolve chromium steels and stainless steels (0·2 g) in $HClO_4$ (4 ml), cool, add HNO_3 (1 + 1, 7 ml), dilute and add ammonium molybdate and Na_2SO_4 soln. (10%, 3 ml) and repeat the extraction with two 10-ml portions of the same solvent.
K. Saito

1746. **Chromatographic determination of vanadium in steel.** I. P. Ryazanov and L. M. Chistota. *Sb. Nauch. Trud. Magnitogorsk. Gorno-Metallurg. Inst.*, 1958 (16), 154–160; *Ref. Zhur., Khim.*, 1958,

(21), Abstr. No. 70,543.—The elution of the peroxide compound of V from a column of chromatographic Al_2O_3 in the presence of $KHCO_3$ was studied. To 1 to 10 ml of a soln. of NH_4VO_3 were added 8 to 10 drops of 3% H_2O_2 and ≃ 25 ml of a saturated soln. of $KHCO_3$, the soln. was heated to decompose H_2O_2, cooled and passed through the column. The eluate and wash liquors were used for the photometric determination of V. It was established that under these conditions there is practically complete elution of NH_4VO_3 (1 to 1·6% of the V remains on the column). The separation of V from Mo [as $(NH_4)_2MoO_4$], W (as Na_2WO_4) and Fe (as $FeCl_3$) was studied. A mixture of soln. of NH_4VO_3 and the second component was treated as described above and V determined photometrically in the eluate. It was established that V is separated from Mo, W and Fe to a satisfactory degree (max. error 2·1%). The described method has been used for the separation of V from Mo, W and Fe in steel and ferrovanadium. Dissolve the sample (0·2 to 0·5 g) in aqua regia, dilute the soln. with hot water (10 to 20 ml), heat for 3 to 5 min., add 2 to 6 ml of $HClO_4$, heat to dense fumes of $HClO_4$ (oxidising Cr to CrO_4^{2-}), add dry NaCl in 0·15 to 0·2-g portions till no more brown fumes of CrO_2Cl_2 are evolved, cool, and dilute to 100 ml. Treat an aliquot as described above to determine V. Reproducible results are obtained. C. D. KOPKIN

1747. **Spectrographic determination of manganese and silicon in ferrous alloys.** A. V. Kozlova and P. D. Korzh. *Sb. Nauch. Trud. Magnitogorsk. Gorno-Metallurg. Inst.*, 1958, (16), 132–136; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,601.—The method is used to determine Mn and Si in ferrous alloys of various compositions from single calibration curves. Carefully grind 0·4 g of graphite, 0·05 g of sample and 0·05 g of $CuCl_2$ for 10 min. in a mortar and place the mixture in a hole in a carbon electrode of 2 mm depth and 1·5 mm diam. (thickness of walls 0·2 mm). Excite the spectra in an a.c. arc discharge at 6 amp. with arc gap 1·5 mm. The exposure is 20 sec. without preliminary ignition. Carry out the analysis on a medium spectrograph from the lines Mn 2939 and Cu 2824 A, and Si 2881 and Cu 3036 A. Construct a calibration curve on the co-ordinates ΔS and log C. No influence of other elements is observed. It is essential that the sample be very finely ground (300 mesh) and that the Cu be introduced only as $CuCl_2$. C. D. KOPKIN

1748. **Spectrographic determination of ferro-alloys. I. Spectrographic determination of a micro amount of arsenic and antimony in ferromanganese.** Yu Yokoyama (Inst. of Iron, Steel and other Metals, Tohoku Univ., Katabira-cho, Sendai). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (7), 812–815.—A stable excitation of the As (0·1 to 0·3%) and Sb (0·03 to 0·06%) lines in ferromanganese is effected by an intermittent a.c. arc (220 V; 10 amp.; interruption factor, 1/3; period, 1/6 sec.; pre-arc, 7 sec.; exposure, 20 sec.) between carbon electrodes. The sample is pulverised and mixed with half its weight of pure carbon powder and one-third of its weight of Ag_2O (internal standard) and placed in a crater (diameter, 2·5 mm; depth, 3 mm) of a carbon rod (diameter, 3·5 mm). By the use of the line pairs Ag 2309·64 - As 2288·12 and Ag 2309·64 - Sb 2311·47, the precisions are 5 to 11% and ≃ 6%, respectively, with a medium spectrograph. K. SAITO

1749. **Use of 8-hydroxyquinoline derivatives for determining certain elements. II. Spectrophotometric determination of cobalt by means of 5:8-quinolinedione dioxime.** A. P. Golovina, I. P. Alimarin and D. I. Kuznetsov (Anal. Chem. Dept., Moscow Univ.). *Vestn. Moskov. Univ.*, 1957, (5), 187–191.—The soln. (5 ml) of Co (0·08 to 7 μg) with a pH of 6·3 to 7·6 (borax buffer) is treated with 5 ml of a 0·04% soln. of the reagent in *iso*-amyl alcohol and the extinction of the alcohol layer obtained after shaking (4 min.) is measured at 416 mμ. The reaction is sensitive to 0·004 μg per ml of Co, and Beer's law is obeyed over the range indicated above. There is no interference by Mn nor by 10,000 times as much Fe. G. S. SMITH

1750. **Study of the conditions of separation of bismuth from cobalt and the polarographic determination of the latter.** A. I. Busev and P. N. Kovalenko (Anal. Chem. Dept., Moscow Univ.). *Vestn. Moskov. Univ.*, 1957, (5), 151–156.—The best conditions for electrolytic separation of Bi (≃ 0·1 g) from Co in a reasonable time (60 min.) from a vol. of 100 ml are found to be deposition on a copper-coated platinum cathode from a soln. ≃ 0·13 *N* in HNO_3 at 70° and 2 V. No potential control is necessary and the Bi is almost completely (98·6%) removed as a dense grey deposit. To determine Co, the soln. after the electrolysis is evaporated to a small vol., treated with KSCN to give a final 0·8 *N* soln., diluted to 50 ml after adjustment of the pH to between 5 and 6, and polarographed to give the height of the cobalt wave ($E_{\frac{1}{2}} = -1·047$ V *vs.* the N.C.E.). With 0·12 to 0·75 mg of Co in the presence of ≃ 1 g of Bi the error is ± 5% of the content. G. S. SMITH

1751. **Spectrophotometric method for the determination of nickel.** L. D. Brake, W. M. McNabb and J. F. Hazel (Dept. of Chem., Univ. of Pennsylvania, Philadelphia, U.S.A.). *Anal. Chim. Acta*, 1958, **19** (1), 39–42.—The stable, coloured complex of EDTA (disodium salt) and nickel was determined in the i.r. band at 1000 mμ where there is no interference from Al^{III}, Be^{II}, Ca^{II}, Cd^{II}, Cr^{III}, Fe^{III}, Hg^{II}, Mn^{II}, Pb^{II}, Sr^{II}, Th^{IV} or Zn^{II}. Interference is caused by Ce^{IV}, Bi^{III}, Ag^{I}, Ba^{II}, Cu^{II}, Co^{II}, Mg^{II} and V^{IV}; Co, Cu and Fe can be separated on an ion-exchange resin column, and Co in mixtures of Co and Ni is masked by nitroso-R salt. The max. deviation is ± 0·10 mg on 20 mg of Ni. F. J. M.

1752. **Determination of iron, cobalt and copper in nickel without taking drillings.** G. V. Loshkareva (S. M. Kirov Ural Polytech. Inst.). *Zavod. Lab.*, 1958, **24** (7), 813–814.—Three drops (0·1 ml) of aqua regia which has been kept for 1 to 2 days are placed on the cleaned surface of the sample. After 10 min. the soln. is transferred by means of a capillary to a tube and washings are transferred similarly. After being boiled and cooled the soln. is diluted to 10 ml for the determination of Fe and Cu by the thiocyanate and the *o*-methoxyphenol (guaiacol) methods. A similar soln. is prepared for the determination of Co by nitroso-R salt. G. S. SMITH

1753. **Determination of intermetallic phases in nickel alloys.** N. M. Popova and A. F. Platonova. *Zavod. Lab.*, 1958, **24** (7), 810–812.—The anolyte is a soln. containing 10 g of $CuSO_4$, 1 g of citric acid and

250 ml of ethanol in one litre, the catholyte is a soln. containing 10 g of $CuSO_4$, 10 g of citric acid and 10 ml of ethanol in one litre, and electrolysis is carried out for from 0·5 to 2 hr. at room temp. with a c.d. of 0·06 amp. per sq. cm. G. S. SMITH

1754. **Counter-current extraction separation of some platinum-group metals.** E. W. Berg and W. Senn, jun. (Coates Chem. Lab., Louisiana State Univ., Baton Rouge, U.S.A.). *Anal. Chim. Acta*, 1958, **19** (1), 12–17 (in English).—Distribution coefficients for the partition of the chloro complexes of Pd, Pt, Rh and Ir between various concn. of HCl and tributyl phosphate were determined and showed high values for Pt and Ir and low values for Pd and Rh. A concn. of 3 *M* HCl gave the optimum difference of distribution value with minimal concn. of reagent. The counter-current technique was carried out by extracting the aq. phase (3 *M* in HCl and satd. with NaCl and tributyl phosphate) with tributyl phosphate. Platinum and palladium were separated almost completely after passage through 16 tubes. Platinum and rhodium required only 5 tubes. F. J. M.

1755. **Separation of fission products by the distillation method. IV. Isolation of carrier-free ruthenium with lead dioxide.** Tomihisa Kambara (Fac. of Educ., Shizuoka Univ., Oiwa-cho, Shizuoka). *Japan Analyst*, 1958, **7** (7), 439–441.—The use of PbO_2 for the oxidation of Ru in fission products was compared with that of other oxidising agents, including $K_2Cr_2O_7$, $Ce(SO_4)_2$ and $(NH_4)_2S_2O_8$. Ruthenium is distilled from 50 ml of 44% (v/v) H_2SO_4 with PbO_2 (2 g). The yield was 97% and the decontamination factor was 2×10^4, after 15 minutes' distillation. K. SAITO

1756. **New aspects of the analytical chemistry of palladium.** L. Martínez Conejero (Laguna Univ., Teneriffe, Canary Isl.). Thesis, Laguna Univ., 1958, 62 pp.—The properties of N'-acetyl-N-phenylselenourea as an analytical reagent for the noble metals have been examined. The reagent is stable in ethanolic soln., and, while giving brown colours with many of the metal ions, it is particularly sensitive for Os^{8+} and Pd^{2+}, 1 μg of the Pd^{2+} in 0·1 ml being detectable. Factors affecting the spectrophotometric determination of Pd by means of the reagent, *e.g.*, pH, temp., concn. and stability of the complex, have been examined. The complex is formed from 1 atom of Pd and 1 mol. of the org. compound and Beer's law is obeyed within the range pH 2·4 to 9·8. The best conditions for the determination are a concn. of Pd of 32 to 320 μg in 25 ml of a slightly ammoniacal aq. ethanolic soln. of pH 9·8, and measurement of the extinction at 470 mμ. Many other metal ions, *e.g.*, Pt^{4+}, Cu^{2+} and Ag^+, interfere, although not reacting sufficiently rapidly or completely to be estimated themselves. The pptn. of Pd by 2-furfuraldoxime (formed *in situ*) has been examined and the following procedure for the determination of Pd developed. The soln. of Pd (30 to 40 mg) in 0·36 *N* HCl (100 ml) is treated with furfuraldehyde (0·5 g), Na acetate (0·25 g in 10 ml of 50% ethanol) and hydroxylamine hydrochloride (0·5 g in 10 ml of methanol). After 3 hr. the ppt. is filtered off, washed with 0·05 *N* HCl and H_2O, dried at 110°, and weighed. The method is specific; the only metals found to interfere were Au and those forming insol. chlorides. L. A. O'NEILL

1757. **Colorimetric determination of palladium with *p*-ethylsulphonylbenzaldehyde thiosemicarbazone.** Sumio Komatsu, Hirohumi Nishimura and Zin Hiroaki (Fac. of Liberal Arts, Toyama Univ., Toyama). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (7), 895–896.—This reagent forms an insol. salt with Pd in aq. soln. (Komatsu *et al.*, *Anal. Abstr.*, 1957, **4**, 2215), but this ppt. (< 30 μg of Pd) dissolves in 30% ethanol and the soln. remains clear for 30 min. The max. absorption at 420 mμ obeys Beer's law for 1 to 7 μg per ml. Interference results from Pt, Au, Ag (> 1·5 μg per ml), Hg (> 9·5 μg) and Cu (> 5·3 μg). K. SAITO

1758. **Determination of the mullite and glassy phase contents of ceramic bodies.** K. Konopicky and E. Köhler (Forschungsinst. der Feuerfest-Industrie, Helmholtzstr. 27, Bonn). *Ber. dtsch. keram. Ges.*, 1958, **35** (6), 187–193.—Extensive trials were made on the chemical attack of HF solution on powdered calcined clay, firebrick, stoneware and porcelain bodies for a range of time and temp. Even though some quartz remained unattacked, some of the mullite had already been dissolved. (The intensity of the mullite X-ray diffraction lines decreased with increase of the time of treatment, an effect which is most pronounced with soft fired bodies.) If therefore a supposedly pure mullite which has been separated with HF is used to standardise the X-ray determination of the mullite content of a sample, the value will be incorrectly high. Except for the most vitrified materials, the glassy phase content is best removed by treatment with 1% HF solution at 0° for 2 hr. The value for the glass content should be checked for its consistency with the mullite, quartz, tridymite and cristobalite contents. The mullite grains are best separated by treatment with 10% HF solution for 2 hr. at room temp. and a correction should be made for its free SiO_2 content (X-ray diffraction). A rapid determination of the mineralogical composition of a sample body by X-ray diffraction should be checked by the separation of the mullite with HF. J. A. SUGDEN

1759. **Spectrographic analysis of opal-type glasses.** W. Ward (Pilkington Bros, St. Helens, England). *J. Soc. Glass Tech.*, 1958, **42**, T240–T247.—Full details are given of a method for determining Fe_2O_3, Al_2O_3, Cr_2O_3, Mn_3O_4, ZnO, CaO, BaO and MgO, which has proved to be very reliable over a period of 6 years. *Procedure*—The sample (1 pt.) is mixed with carbon powder containing the internal standard (CoO 0·4, $SrCl_2$ 5, and CdO 0·75%) (3 pt.) and Ag_2O as emission "buffer" (1 pt.) and is excited by d.c. in a hollow cathode. A series of standard glasses are used for calibration with the line pairs Fe 3021·07 and 2929·16 - Co 2989·6, Cr 2975·5 - Co 2989·6, Mn 2933·1 - Co 2989·6, Zn 3345·0 - Cd 3261·1, Al 2652·5 and 3082·2 - Sr 2931·8, Ca 3006·9 - Sr 2931·8, Ba 3071·6 - Sr 2931·8, Mg 2782·97 - Sr 2931·8. The results agreed within 0·1% with those of chemical methods. J. A. SUGDEN

1760. **Rapid quantitative methods for determination of brighteners, levellers and anti-pitting agents in nickel electroplating baths.** K. Szmidt, T. Zak and Z. Kwiatkowski (Electroplating Div. of Inst. Mech. Precyzyjnej., Warsaw, Poland). *Trans. Inst. Met. Finish.*, 1958, **36** (1), 17–21.—Rapid and accurate methods, based on polarography and surface-tension measurements, are reported for

determination of certain brightening and wetting agents in nickel electroplating, with special reference to Cd. C. H. COWPER-COLES

See also Abstracts—**1602**, Applications of some org. comp. of Se. **1603**, Pptn. of the iron and titanium groups with tannic acid. **1604**, Hexamine in quant. analysis. **1605**, Starch - iodine as an indicator in arsenometric and bromatometric titrations. **1607**, **1608**, **1609**, **1611**, Determination of As^{3+}. **1609**, Oxidation with NaClO and ICl. **1610**, Oxidation with NaClO. **1611**, Oxidation with $K_2S_2O_8$ and ICl. **1613**, Oxidation with $Fe(CN)_6^{3-}$ and OsO_4. **1614**, New titrimetric methods by pptn. **1615**, Use of 1:2-diamino*cyclo*hexanetetra-acetic acid in complexometry. **1821**, Determination of As in petroleum. **1823**, Determination of C and H in coal. **1848**, Histochemical determination of K. **1849**, Determination of ^{137}Cs in foods, etc. **1850**, Determination of ^{89}Sr and ^{90}Sr in vegetation, etc. **1851**, Determination of Th in plants, etc. **1852**, Determination of ^{59}Fe in biological systems. **1853**, Determination of Co and Cu in biological material. **1931**, Determination of ions in fermented liquids. **1937**, Detection of trace metals in fats. **1952**, Colorimetric methods for determination of P. **1971**, Chromatographic separation of some cations. **1976**, Gas-chromatographic analysis of gas mixtures.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives.

1761. **Contribution to the detection of organic compounds. Colour reactions of unsaturated compounds. II.** L. Rosenthaler (Seftigenstrasse 40, Bern). *Pharm. Acta Helv.*, 1958, **33** (7), 269–273.—The colour reactions given by a number of unsaturated compounds with the reagents named are listed. Some are new, others are re-investigations of previously reported work. The compounds reacting positively are mostly of natural origin, *e.g.*, citral, oleic acid, eugenol, terpineol, anethole, and analogues, though vinyl acetate and vinyl ether also react. In general, 0·05 g in aq. soln. is used, unless the compound is insoluble, when a little ethanol is added. *Reagents*—Sodium nitroprusside and alkali; *m*-dinitrobenzene and alkali; 4-chloro-1:3-dinitrobenzene and alkali; alkaline soln. of *m*-dinitrobenzoic acid; phloroglucinol-conc. HCl; pyrrole - conc. HCl; indole - conc. HCl. Only for indole - HCl are specific experimental details given, *viz*, 1 ml of a glacial acetic acid soln. of the unsaturated compound is mixed with 5 drops of 1% indole in glacial acetic acid and 2 ml of conc. HCl. Similarities between the colour reactions of unsaturated compounds and aldehydes are discussed, and it is shown that the colours obtained with, for example, Schiff's reagent or *m*-phenylenediamine - dil. H_3PO_4, are not due to contamination with aldehydes. H. M.

1762. **Simplicity in organic micro-analysis.** T. S. West. *Chem. Age*, 1958, **80**, 193–194.—Recent methods for the simple and rapid decomposition of organic compounds for elementary analysis are reviewed. N. E.

1763. **Swift combustion of organic compounds for analysis.** I. K. H. Otter (Fisons Pest Control, Ltd., Chesterford Park, Essex, England). *Nature*, 1958, **182**, 393.—The all-glass apparatus described and illustrated operates as a closed system, permitting the rapid combustion of the sample (3 to 7 mg, solid or liquid) and collection of the products in the absorption tube. The contents of this tube can then be transferred to a container according to the analysis required, *e.g.*, halogens, S or P. W. J. BAKER

1764. **An improved method for the Körbl micro-determination of carbon and hydrogen.** E. Pella (Ist. Carlo Erba per Ricerche Terapeutiche, Milan, Italy). *Mikrochim. Acta*, 1958, (5), 687–695.—Modifications are proposed to Körbl's method (*Mikrochim. Acta*, 1956, 1705) to eliminate the high blanks for C and H. The effectiveness of the Körbl catalyst (silver permanganate) is confirmed, but it is emphasised that the use of such an energetic catalyst, to achieve rapid combustion, necessitates strict adherence to analytical conditions if low blanks are to be maintained. Under ideal conditions the method is capable of a precision of ± 0·1%. D. F. PHILLIPS

1765. **Vacuum determination of carbon and hydrogen in organic substances containing sulphur and halogens.** P. N. Fedoseev and L. S. Ignatenko. *Izv. Akad. Nauk TurkmenSSR*, 1957, (6), 84–89; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,617.—The determination of C and H by the empty-tube method (*Anal. Abstr.*, 1958, **5**, 3764) is applied to substances containing S and a halogen. The substance is ignited in the presence of CuO and the halogen and oxides of S are absorbed by metallic silver heated to 750° or 800°. Introduce into the ignition tube a silver spiral (70 to 80 mm), a layer of CuO as wire (130 to 140 mm), a boat (120 to 140 mm) with silver chips or foil (8 to 10 g) and a spiral of CuO in which is inserted a spiral of silver wire. Both ends of the ignition tube and the exit tube are fitted with screw clips; the exit is connected to vacuum. The tube is heated with three electric furnaces, reaching 750° or 800°. Place the sample (0·03 to 0·06 g) between the boat and the copper oxide spiral, close the clips and exhaust the tube to a pressure of 3 to 5 mm. Close the clip on the exit tube and rapidly move a furnace from the boat to the sample. Heat the whole of the tube for 8 to 12 min., open the clips on the ends of the tube and draw out the products with a stream of O. The error of the determination for H is ± 0·20% and for C ± 0·30%. A new apparatus is proposed for the absorption of the H_2O and CO_2. C. D. KOPKIN

1766. **Micro-determination of carbon and hydrogen in organic compounds containing selenium.** A. S. Zabrodina and S. Ya. Levina (Org. Chem. Dept., Moscow Univ.). *Vestn. Moskov. Univ.*, 1957, (5), 181–186.—Combustion in O in an empty tube with the use of finely ground quartz as a filter to retain SeO_2 is described. Results of determinations of C and H in a large number of organic selenium compounds are given. The error is ≯ ± 0·3% of the sample wt. G. S. SMITH

1767. **Methods of combustion in a sealed tube for quantitative ultra-micro elementary organic analysis.** W. Kirsten (Inst. Med. Chem., Univ. Uppsala, Sweden). *Chim. Anal.*, 1958, **40** (7), 253–263.—After recapitulation of usual methods for the determination of S, N and P, using open and sealed

tubes, a method is described for the micro-determination of C, H and N on the same sample, by combustion in an oxygen-filled sealed tube in the presence of metallic copper at 700° for 1 hr. The sealed tube is introduced into a special apparatus, which is evacuated, the sealed tube is broken, and the sum of N, CO_2 and H_2O is measured by the pressure developed within the apparatus. The volume of gas is then measured at atmospheric temp. and pressure and, when corrected for the partial pressure of the water vapour, gives the sum of N and CO_2. Finally, the CO_2 is absorbed with KOH and the residual N measured. This procedure was satisfactory for samples also containing halogens and P, but gave low results for H with samples containing S, probably owing to formation of $CuSO_4$ and retention of moisture thereon. This was overcome by adding lead dichromate to the sealed tube. The successive improvements in apparatus and technique are described, culminating in the production of an apparatus (illustrated) for handling samples of 0·1 mg, used in conjunction with an ultramicro-balance. After the determination of C, H and N in this apparatus, the broken tube, containing any S, P and halogens combined with lead dichromate, can be recovered, making possible a complete analysis for C, H, N, S, P and halogens on a single sample weighing 0·1 mg.

R. E. ESSERY

1768. **The preparation of silver dichromate and its use to produce a catalyst for carbon and hydrogen analysis by combustion.** E. Ruf (Anal. Lab., Th. Goldschmidt A.-G., Essen, Germany). *Z. anal. Chem.*, 1958, **163** (1), 21–27.—Two forms of silver dichromate are described and are prepared in the following manner. (*a*) Dissolve $AgNO_3$ (25 g) in 4 litres of water. Add conc. HNO_3 (15 ml) followed by a cold soln. of $K_2Cr_2O_7$ (66 g) and conc. HNO_3 (10 ml) in 1 litre of water. After several hours, filter off the ppt., wash 3 times with dilute HNO_3 (pH 1·5, 30 ml) and dry at 120° for 1 hr. (*b*) Dissolve $K_2Cr_2O_7$ (87 g) in 4·75 litres of water; add conc. HNO_3 (20 ml) and a cold soln. of $AgNO_3$ (50 g) in 0·25 litre of water. Filter off, wash and dry the ppt. as in (*a*). By heating for 30 min. at 400° and for 2 hr. at 950° both forms are converted into oxidation catalysts which function as well as the more commonly used ones and can be used repeatedly without losing efficiency. Comparisons are given of the analysis of benzoic acid using the silver dichromate preparations and a number of other catalysts. W. T. CARTER

1769. **A new type of combustion tube for carbon - hydrogen determination by the "rapid combustion method."** W. M. Hazenberg (Univ. Gronigen, Netherlands). *Mikrochim. Acta*, 1958, (5), 709–712 (in English).—A tube maintaining classical horizontal combustion, but used in conjunction with the "empty tube" technique of Ingram, is described. Although the apparatus has the normal length, the combustion zone is twice as long as before. Halogen and sulphur oxides are absorbed by "electrolytic" silver wool. The nitrogen oxides are absorbed by MnO_2 in a slightly modified absorber.

D. F. PHILLIPS

1770. **Succinyl chloride as a hydrolytic reagent for the determination of water.** R. Belcher, J. H. Thompson and T. S. West (Univ., Birmingham, England). *Anal. Chim. Acta*, 1958, **19** (2), 148–153.—In the micro-determination of H in organic substances a titrimetric method for the determination of the water formed could offer advantages over the method of direct weighing. Most reagents proposed for this purpose react with water to produce HCl and/or CO_2 which are evolved as gases and can be absorbed and titrated. These methods are briefly reviewed and a number of such reagents are tested in a conventional absorption train, $BaCl_2.2H_2O$ being used as a source of water. Succinyl chloride is a satisfactory reagent for the purpose in question. H. N. S.

1771. **Direct determination of oxygen in organic material.** J. P. Dixon (Shell Research Ltd., Thornton Res. Centre, Chester, England). *Anal. Chim. Acta*, 1958, **19** (2), 141–147.—The difficulties associated with the Unterzaucher method are accentuated when it is applied to petroleum fractions that yield a high proportion of H in the pyrolysis gases and to compounds that contain S. In this modification the O is converted to CO in the usual manner, but the CO is oxidised to CO_2 with CuO instead of I_2O_5. The CO_2 is determined by absorption. Sulphur compounds, H_2S, CS_2 and COS, are removed by passing the gases over heated copper wire prepared by the reduction of copper oxide wire with H. H. N. S.

1772. **Determination of alkoxyl groups in organosilicon compounds.** G. D. Nessonova and E. K. Pogosyants (Moscow Textile Inst.). *Zavod. Lab.*, 1958, **24** (8), 953.—The stability of the iodising mixture used by Kirsten and Ehrlich-Rogozinsky (*cf. Anal. Abstr.*, 1955, **2**, 3080) is confirmed and its use in place of HI for determining ethoxyl groups in tetraethoxysilane is recommended.

G. S. SMITH

1773. **Determination of methoxyl and ethoxyl groups in organic compounds.** L. Filipović and Z. Štefanac (Chem. Lab., Fac. Sci., Univ. Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (2), 149–156 (in English).—Methyl or ethyl iodide is liberated by reaction of the ether with HI (sp. gr. 1·7) containing a little phenol and acetic anhydride to aid dissolution of the sample. Nitrogen is used to flush the apparatus, and the alkyl iodide is purified by passage over ascarite instead of through the customary liquid washers. The iodide is absorbed in a mixture of bromine, acetic acid and Na acetate, excess of bromine is destroyed with formic acid, and the iodate formed is used to liberate iodine from KI in dil. acid soln. This iodine is titrated with 0·02 *N* $Na_2S_2O_3$. Blanks are only about 0·22 ml and a sample weight of 3 to 7 mg is used. The advantages of ascarite are (*a*) the same tube can be used for several determinations without refilling, (*b*) flow rates need not be closely controlled, (*c*) complete removal of interfering substances is achieved and no retention of alkyl halide occurs. In addition, by adding a layer of anhydrone the dried halide can be passed directly into an apparatus for the determination of C and H, but 10 to 20 mg of sample is needed. H. M.

1774. **Combined colorimetric and chromatographic ultramicro-analysis of gases. II. Determination of hydrocarbons.** J. Juránek (Res. Inst. Petroleum, Brno, Czechoslovakia). *Chem. Listy*, 1958, **52** (7), 1289–1298.—The method developed is suitable for the determination of hydrocarbons present in gases in the concn. range 10^{-3} to 10^{-7} % by vol. For the chromatographic analysis of relatively large vol.

of gases, the characteristic elution vol. of hydrocarbons have been studied, use being made of the value of the max. elution vol. of the inert component of the gas. The unsuitability of the use of const. temp. of adsorbent for the ultramicroanalysis of hydrocarbons in gas has been proved. The stability of the colorimetric indication system towards the temp. changes of the adsorption and desorption of hydrocarbons in chromatographic columns has been used in the procedure described, and 2×10^{-8} ml of methane, 1×10^{-5} ml of ethane and 1×10^{-6} ml of higher hydrocarbons in 0·1 to 1000 ml of gas were thus determined. The method has been tested on various sorts of material.
J. ZÝKA

1775. **Determination of propylene in ethylene and ethane - ethylene fractions.** K. A. Gol'bert and A. V. Alekseeva (Sci. Res. Inst. of Synthetic Alcohols and Organic Products). *Zavod. Lab.*, 1958, **24** (6), 688–690.—A gas-chromatographic method for determining concn. of propylene of $\simeq$ 0·2% with a relative error of 10 to 15% is described. G. S. SMITH

1776. **A rapid determination of vinyl chloride vapour in ammonia by means of a detector tube.** Yoshitaka Kobayashi (Fac. of Engng, Yokohama Univ., Minami-ku). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (9), 1140–1143.—Whilst NH_3 is firmly adsorbed by silica gel containing $CoCl_2$, vinyl chloride (**I**) vapour on the gel is readily desorbed by air at room temp. The outlet and the carrier gas are diluted and a portion of the issuing gas is passed through a column of silica gel containing H_2SO_4 and CrO_3. The length of dark-brown layer varies as the amount of **I** (0·05 to 4·0%), which is determined on an empirical diagram. Interference results from unsaturated hydrocarbons, H_2S and SO_2. The error is $<$ 10% and the time for an estimation is $\simeq$ 5 min. K. SAITO

1777. **New determination of "diphosgene."** P. Malatesta, G. Cogliati and G. Migliaccio (Univ. Rome, Italy). *Ric. Sci.*, 1958, **28** (8), 1683–1686.—The Schönemann colour reaction, using *o*-dianisidine or indole, is employed for the determination of trichloromethyl chloroformate ("diphosgene"). Beer's law is observed for 2 to 10 μg of the gas, at 450 mμ, using *o*-dianisidine, and for 40 to 120 μg, at 630 mμ, using indole. C. A. FINCH

1778. **Identification of organic compounds. XXV. Identification and separation of aliphatic C_1 to C_{18} alcohols by paper chromatography.** J. Borecký, J. Gasparič and M. Večeřa (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1958, **52** (7), 1283–1288.—The chromatographic separation of the C_1 to C_{18} alcohols as their 3:5-dinitrobenzoates was studied. Paper impregnated with liquid paraffin and formamide or dimethylformamide as a component of the mobile phase were found to be suitable. *Procedure*—Dissolve the sample (0·1 g) in benzene (1 ml), add a soln. of 0·5 g of 3:5-dinitrobenzoyl-chloride in 3 ml of benzene and 1 ml of pyridine and heat for 30 min. on a water bath. Cool, and shake twice with KOH soln. (50%) (5 ml); after each shaking add 50 ml of H_2O to separate the layers. Extract the benzene soln. with H_2O (2 $\times$ 5 ml), with HCl (1:1) (2 $\times$ 5 ml) and with H_2O (3 $\times$ 5 ml). Add Na_2SO_4. Develop by descending chromatography and dry the chromatogram at 120° to 150°. The spots can be detected by illumination with unfiltered light from a mercury vapour lamp. The esters formed can be observed as dark spots in filtered u.v. light. The detection can also be carried out by reduction of the nitro group to the amino group with $SnCl_2$ soln. and by addition of *p*-dimethylaminobenzaldehyde. Several different combinations for the mobile phase were studied. J. ZÝKA

1779. **The spectrophotometric determination of methanol with chromotropic acid.** M. J. Maurice and B. Veen (Zentr. Anal. Lab. N.V. Onderzoek. Inst. "Research," Arnhem, Netherlands). *Z. anal. Chem.*, 1958, **163** (1), 13–21.—The reaction conditions of Deckenbrock and Sprick (*Branntweinwirtschaft*, 1953, **75**, 1) were used to examine the effect of different sources of reagent and different ages of reagent solution on the accuracy of the determination of methanol with chromotropic acid. An analysis of variance was carried out on 36 duplicate determinations using chromotropic acid from 3 different sources in solutions 1, 3, 7 and 9 days old. It was concluded that the reagent solution could be used for several days and that there was no significant variation between reagents from different sources. W. T. CARTER

1780. **Continuous measurement of the concentration of methanol and ethanol in a binary mixture with water by means of a high-frequency apparatus.** Soichiro Musha and Masami Takeda (Fac. of Engng, Univ. of Osaka Prefecture, Sakai). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (9), 1143–1147.—The authors' high-frequency apparatus (*Ibid.*, 1955, **58**, 482, 486) was applied to continuous measurement. Change in current due to the change in conductivity of the binary system, water - alcohol, is recorded with an automatic balancing recorder. The current varies as the concn. of ethanol and methanol, the coeff. of variation in the standard curve being $\pm$ 0·35% and $\pm$ 1·69%, respectively. The apparatus is calibrated for three ranges, $<$ 50%, 45 to 80%, and 75 to 100% of ethanol. Temp. seriously affects the results; instrumental correction for this was unsuccessful. The temp. of the water introduced into the apparatus must be constant within $\pm$ 0·2°. The relative error for unknown samples is $-$ 3·3 to + 8·5% for methanol and $-$ 2·0 to + 0·4% for ethanol. K. SAITO

1781. **Determination of condensates of ethylene oxide (polyglycols) by precipitation with tungstophosphoric acid.** H. Étienne. *Parfums, Cosmet., Savons*, 1957, (137), 33–37.—The ppt. produced by the action of tungstophosphoric acid on ethylene oxide is converted into an insol. complex in an HCl medium in the presence of $BaCl_2$. This is filtered off and dried under vacuum. The loss in wt. on subsequent calcination represents the amount of ethylene oxide complexed and pptd. This procedure eliminates the influence of aliphatic alcohols when present even in large amounts. The minimum experimental error is $-$ 0·2% (relative) and the max. error 1 to 1·5% (relative). K. R. C.

1782. **A comparison of a glucose oxidase method with an official method for the determination of fructose.** E. F. Potter and K. T. Williams (Western Reg. Res. Lab., Albany, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 681–682.—The method of Williams and Potter (*cf. Ibid.*, 1958, **41**, 307) for the determination of fructose has been compared with that of Jackson and Matthews ("Official Methods of Analysis," A.O.A.C., 8th Ed., 1955, 29·64 and 29·65). In general the

results agree well within experimental error. In the former method, reducing sugars other than glucose and fructose, and non-sugar reducing substances, are included in the fructose value; in the latter method, the extent of inclusion of such substances depends on the experimental conditions. It is immaterial whether clarification is effected by lead or ion-exchange resin. A. A. ELDRIDGE

1783. **Removal of electrolytes from sugar solutions.** R. C. Hughes and W. J. Whelan (Lister Inst. of Prev. Med., London, England). *Chem. & Ind.*, 1958, (28), 884–885.—A special grade of activated charcoal (Ultrasorb S.C. 120/240) adsorbs monosaccharides but not salts. After passage of a mixture through a column of the charcoal, salts are removed by washing with water and sugars are then eluted with aq. ethanol, the minimum dilution for rapid removal of sugars being 0·5% v/v. Borate buffers do not noticeably accelerate the desorption of glucose. Results for glucose, galactose, xylose and ribose, alone and with various salts and buffers, on a large scale with 25% ethanol and a small scale with 5% ethanol, show recoveries from 94 to 109%. Tracer experiments with labelled glucose showed that no sugar was eluted by the amount of water required to remove salts. The charcoal contains alkaline water-soluble matter which can be removed by washing. Its adsorptive capacity for glucose is of the order of 0·6 g of sugar per 100 g of charcoal, the capacity for maltotriose and maltotetraose being at least thirty times this. R. E. ESSERY

1784. **Quantitative determination of amylose in starch by the "blue-value" method using the B. Lange Universal colorimeter.** M. Ulmann and S. Augustat (Inst. für Ernährung Dtsch. Akad. Wiss., Potsdam-Rehbrücke, Germany). *Z. anal. Chem.*, 1958, **162** (5), 337–344.—The photometric procedure described is reasonably rapid and obviates the use of elaborate apparatus. It is based on the separation and determination of amylose by the method of McCready and Hassid (*J. Amer. Chem. Soc.*, 1943, **65**, 1154), but the Lange colorimeter is used to measure the extinction of the brilliant blue soln. The prep. of the sample soln., the construction of the calibration curve and the operation of the instrument are given. An OG2 filter (644 to 700 mμ) and a 100-ml cell (34·5 mm depth) ensure max. accuracy (within ± 2%). W. J. BAKER

1785. **Determination of aldehydes with 2:4-dinitrophenylhydrazine.** Senjiro Maruta and Yoshihito Suzuki (Chem. Dept., Fac. Liberal Arts, Yamanashi Univ., Kofuchu-machi, Kofu). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (9), 1147–1149.—Paper chromatography of 2:4-dinitrophenylhydrazones of various aliphatic aldehydes was studied with several developing agents on filter-paper containing different fixed phases. The R_F value decreases with increasing length of carbon chain. For the separation of spots of lower aldehydes, a mixture of methanol and light petroleum (boiling-range 140° to 160°) (20:3) is the best developing agent and a hydrocarbon is the best fixed phase. The R_F value decreases slightly with increase in amount (0·025 to 0·3 g per 100 sq. cm) of the fixed phase, whilst the time taken for development increases. The spot is cut out, extracted with ethanol and submitted to photometry at 440 mμ. The max. absorption remains unchanged for butyl and lower aldehydes. The method is applicable to the determination of aldehyde in wine. *Procedure*—Dilute the sample (1 g) to 10 ml with ethanol containing 2:4-dinitrophenylhydrazine (1 mg per ml) and 2 *N* HCl. Wet the paper with ligroin (boiling-range 120° to 140°) (0·17 g per 100 sq. cm) and spot the sample soln. (10 mg) on it. Develop with the mixture until the developer ascends 20 cm. Cut off the two spots corresponding to acetaldehyde (R_F 0·69) and *n*-butyraldehyde (0·66); extract each with ethanol (10 ml) and submit to photometry. K. SAITO

1786. **Simultaneous quantitative analysis of formaldehyde and polyoxymethylene.** Z. Bellen (Inst. Gen. Chem., Warsaw, Poland). *Chim. Anal.*, 1958, **40** (7), 250–253.—Free formaldehyde is determined by condensation with dimedone and titration of the complex, and the sum of aldehyde and polyoxymethylene by oxidation with excess of iodine in alkaline soln. and back-titration. For the latter, a sample containing 20 to 40 mg is placed in a glass-stoppered flask, and treated with water (10 ml), 0·1 *N* iodine (50 ml) and *N* NaOH (50 ml). After shaking the mixture and setting it aside in the dark for 10 min., *N* H_2SO_4 (55 ml) is added and the excess of iodine is titrated with 0·1 *N* $Na_2S_2O_3$, with starch as indicator. For free formaldehyde, a sample containing 7·5 to 12 mg is placed in a glass-stoppered flask, and treated with aq. dimedone soln. (50 ml, concn. not given) and acetate buffer (70 ml, pH 4·6). After being set aside for 3 hr. with occasional shaking, the liquid is filtered on a fritted glass filter, and the ppt. is washed with cold water (10 × 5 ml), and then washed into a flask with ethanol, which dissolves the complex, but leaves the polyoxymethylene in suspension. This soln. is titrated with 0·1 *N* NaOH to a pink colour with phenolphthalein, stable for 1 min., or potentiometrically, with glass - calomel or antimony - calomel electrode systems. R. E. ESSERY

1787. **Cerate - chromate oxidimetry. III. Estimation of formaldehyde and formic acid, and of formic and acetic acids, in their mixtures.** N. N. Sharma (Univ. of Allahabad, India). *Z. anal Chem.*, 1958, **162** (5), 321–324 (in English).—A mixture of formic acid (**I**) and formaldehyde (**II**) can be analysed by heating the sample (5 ml) under reflux for ≃ 5 min. with standard $Ce(SO_4)_2$ soln. (10 ml) in dil. H_2SO_4. This converts **II** into **I**; the excess of $Ce(SO_4)_2$ can be determined with standard Fe^{II} soln. A duplicate sample is heated under reflux for 5 min. as described above, after which conc. H_2SO_4 plus a few drops of a 1% (w/v) soln. of chrome alum are added and refluxing is continued for 60 to 90 min. to oxidise both **I** and **II** completely to CO_2 plus H_2O. The content of **II** can then be calculated from the total $Ce(SO_4)_2$ consumed minus the $Ce(SO_4)_2$ consumed by **I**. Because cerate - chromate reagent has no effect on acetic acid (**III**), a mixture of **I** and **III** can be analysed by titrating the total acid with standard NaOH soln. and then determining the concn. of **I** by oxidation with $Ce(SO_4)_2$ in the presence of Cr^{3+} and conc. H_2SO_4. Results are satisfactory. W. J. BAKER

1788. **Solubilisation chromatography. II. Ketones.** J. Sherma and W. Rieman, III (State Univ., New Brunswick, N.J., U.S.A.). *Anal. Chim. Acta*, 1958, **19** (2), 134–140 (*cf. Anal. Chim. Acta*, 1958, **18**, 214).—A study is made of the elution of ketones through ion-exchange resins with aq. soln. of acetic acid or the lower alcohols as eluents. A

mixture of seven ketones, the highest being undecan-2-one, is separated by this technique. The elution of ketones with aq. soln. of a salting-in salt is also studied. H. N. S.

1789. **Ion-exchange chromatography (of organic acids).** D. V. Myhre and F. Smith (Univ., Minnesota). *J. Org. Chem.*, 1958, **23** (8), 1229–1230.—This paper deals with the separation of organic acids by using papers containing ion-exchange resins and O-(carboxymethyl)cellulose in conjunction with non-acidic solvents. The papers are prepared by adding the powdered ion-exchange resin to the cellulose-pulp before the sheets are made. Strongly acidic resin papers are useful for the separation of gluconic and glucuronic acids. Weakly acidic papers are most useful for separating amino acids, but weakly basic papers are of no value in this respect. Filter-paper impregnated with O-(carboxymethyl)cellulose gave a good separation of uronic acids and lactones. K. R. C.

1790. **Conductimetric titration of carboxylic and phenolic acids in non-aqueous solutions. I. Preliminary experiments.** N. van Meurs and E. A. M. F. Dahmen (Koninklijke/Shell-Laboratorium, N. V. De Bataafsche Petroleum Maatschappij, Amsterdam, The Netherlands). *Anal. Chim. Acta*, 1958, **19** (1), 64–73.—The bases used as titrants were K methoxide (**I**) in pyridine - benzene and tetramethylammonium hydroxide (**II**) in methanol - benzene, both at $\simeq 0{\cdot}3\ N$. Solvents for the acids were methanol, pyridine and dimethylformamide (**III**). Resistance measurements were made with a Philips conductivity bridge after each 0·1-ml addition of titrant. The best results were given by the following base - solvent combinations—for aliphatic monocarboxylic acids, **II** and **III**; aromatic monocarboxylic acids, **I** and **III**; aliphatic dicarboxylic acids, **I** and **III** or methanol; aromatic dicarboxylic acids, **II** and pyridine - **III** mixture; alkylphenols and derived phenols, **I** and pyridine. F. J. M.

1791. **The ferric hydroxamate colour reaction. II. The spectrophotometric determination of acetic anhydride.** K. J. Morgan (Chemical Defence Exp. Estab., Porton, Wilts., England). *Anal. Chim. Acta*, 1958, **19** (1), 27–31 (in English).—The observed optical density of the ferric acetohydroxamate formed during the reaction between hydroxylamine, $HClO_4$, acetic anhydride and $FeCl_3$ in the determination of hydroxyl groups (*cf.* Baritrop and Morgan, *Anal. Abstr.*, 1958, **5**, 563) was found to be very sensitive to variations in the time factors in the sequence of operations. Under careful control of these factors, the results were reproducible and the colour reaction was found to obey Beer's law up to a concn. of $5 \times 10^{-5}\ M$. F. J. M.

1792. **Assay of tributyl phosphate in organic solvents.** U. A. Sant and H. Sankar Das (Atomic Energy Estab., Bombay). *Anal. Chim. Acta*, 1958, **19** (2), 202–203.—Tributyl phosphate is used in the extraction of compounds of uranium, and the necessity for its determination in organic diluents, such as kerosine, arises. *Procedure*—To 2 ml of the sample, in a nickel crucible, add sufficient powdered NaOH to absorb it, and cover the mixture with more NaOH. Cover the crucible and heat very slowly so that in $\simeq 1$ hr. the mass becomes a homogeneous liquid. Dissolve the cooled mass in dil. $HClO_4$, dilute to 250 ml, neutralise a 100-ml aliquot to litmus, add 6% (v/v) $HClO_4$ ($\simeq 4$ ml) and titrate with standard bismuthyl perchlorate soln. with the use of a saturated soln. of di(allylthiocarbamoyl)hydrazine in $CHCl_3$ as extraction indicator (*cf. Anal. Abstr.*, 1955, **2**, 1150). H. N. S.

1793. **Polarographic determination of primary aliphatic amines.** N. F. Alekseev (Kemerovo Nitro-Fertiliser Works). *Zavod. Lab.*, 1958, **24** (6), 684–688.—Compounds of primary aliphatic amines with *iso*butyraldehyde of mol. ratios 1:2 and 1:1 in $0{\cdot}1\ N$ LiCl give reproducible polarographic waves on the dropping mercury electrode at $E_{\frac{1}{2}} = -1{\cdot}5$ V. The aldehyde itself is reduced at $-2{\cdot}1$ V. Secondary and tertiary amines and NH_3 do not interfere. G. S. Smith

1794. **Potentiometric determination of vinyl cyanide by means of hydroxylamine.** A. P. Terent'ev, S. I. Obtemperanskaya and M. M. Buzlanova (M. V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1958, **24** (7), 814–815.—The sample (0·6 to 1·0 g) is placed in an ampoule which is broken under the surface of a mixture of 25 ml of dioxan and 25 ml of $8\ N$ hydroxylamine prepared by neutralisation of a soln. of hydroxyammonium chloride with NaOH soln. (first with 40% and then with $0{\cdot}1\ N$), and the soln. is diluted to 100 ml with $8\ N$ hydroxylamine. The reaction gives β-hydroxyaminopropionitrile. After 12 hr. a 10-ml aliquot is treated with 10 ml of acetone to combine with the free hydroxylamine and, after 10 min., with 10 ml of water. The soln. is titrated potentiometrically (glass electrode and N.C.E.) with $0{\cdot}1\ N$ HCl. G. S. Smith

1795. **Identification of organic compounds. XXIV. Separation and identification of sulphides by paper chromatography.** J. Petránek and M. Večeřa (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1958, **52** (7), 1279–1282.—Sulphides, when treated with the sodium salt of *p*-nitrobenzenesulphonchloroamide (**I**), give the corresponding *p*-nitrobenzenesulphonylsulphilimines, which can be separated by paper chromatography. *Procedure*—To a soln. of 0·5 millimole of sulphide in 2 ml of methanol add a soln. of **I** (200 mg in 5 ml of methanol). After 15 min. dilute the decolorised soln. with H_2O (20 ml), add $2\ N$ NaOH (10 ml) and extract twice with $CHCl_3$ (5 ml). Place the $CHCl_3$ extract (corresponding to 1 to 10 μg of sulphilimine) on Whatman No. 4 paper impregnated with a 10% soln. of formamide in ethanol, and develop with the use of descending chromatography at 20° to 21°, with benzene or a mixture of benzene - *cyclo*hexane (1:1 or 3:2) as mobile phase. Moisten the chromatogram with $SnCl_2$ soln. (0·7 g of $SnCl_2$ in 100 ml of 15% HCl) and set aside for 30 min. For the detection use a 1% soln. of *p*-dimethylaminobenzaldehyde in ethanol containing 5% HCl; yellow spots appear. The described procedure is suitable for the separation of homologues of dialkyl and benzyl alkyl sulphides and can also be used for the separation of alkyl aryl and diaryl sulphides. R_F values of *p*-nitrobenzenesulphonylsulphilimines of 12 aliphatic sulphides and of 10 aromatic benzyl and aryl alkyl sulphides are given. The preparation of **I** is described. J. Zýka

1796. **The determination of methylenebisthioglycollic acid.** A. Miss and A. Arsene. *Rev. Chim., Bucharest*, 1958, **9** (6), 331–332.—The method is based on the esterification of the acid with an alcohol which is almost immiscible with water, having a b.p. sufficiently low for the inorganic acids

present not to destroy the organic substances, nor to form esters with inorganic acids, especially H_2SO_4. *Procedure*—Neutral or alkaline soln. are acidified to pH 1 to 2 to liberate the methylenebisthioglycollic acid (**I**), and the procedure varies according to the concn. of **I**. (*A*) For soln. with $> 1\%$ of **I** and weak in inorganic acids and salts, a vol. containing $\simeq 1$ g of **I** is placed in a distillation flask together with *n*-butanol (30 ml) and a drop of H_2SO_4 and distilled until no more water is carried over, and continued for another 15 min. On cooling, 20 ml of ethanol is added with 3 drops of metacresol purple soln. and the product is neutralised with 0·5 *N* NaOH and a further 20 ml is added. After saponification for 30 min. and cooling, it is titrated with 0·5 *N* HCl. (*B*) When the percentage of **I** is low, and the soln. is rich in inorganic salts and acids, a liquid - liquid extractor is used to concentrate the organic acid, and the procedure is then as above. The max. error is claimed to be 0·8%.

H. Sher

1797. **Quantitative determination of dithiocarbamates and thiuram disulphides.** C. L. Hilton and J. E. Newell (U.S. Rubber Co., Wayne, N.J.). *Rubb. Age*, 1958, **83** (6), 981–984.—Zinc dithiocarbamates and thiuram disulphides are extracted from rubber with ethanol, and separately determined. The dithiocarbamates are decomposed by H_3PO_4 in ethanol, the resulting CS_2 is distilled into diemthylamine - ethanol and the absorption at 287 mμ measured. The thiuram sulphides remaining in the extracting solvent are decomposed with $NaHSO_3$ after neutralisation with NaOH, the distillation and collection are repeated, and the absorption at 285 mμ is measured immediately distillation is completed. Shredded rubber (0·5 g) is extracted at the b.p. with 95% ethanol (400 ml) containing 85% H_3PO_4 (1 ml) and distilled into a mixture of 5% aq. dimethylamine soln. (5 ml) and 95% ethanol (15 ml), to a total volume of 250 ml. The absorption at 287 mμ is measured. The residue is neutralised with 100 ml of 0·1 *N* NaOH, 100 ml of a 0·1% aq. $NaHSO_3$ soln. and 50 ml of 95% ethanol are added, and the distillation is repeated. For the Zn dialkyl- or diaryl-dithiocarbamates, the percentage content is given by absorption at 287 mμ × 0·55 (methyl), × 0·65 (ethyl), × 0·85 (butyl) and × 1·10 (benzyl), and × 0·69 for Zn pentamethylenedithiocarbamate. For the tetra-alkylthiuram sulphides, the percentage content is given by absorption at 285 mμ × 0·438 (methyl) and × 0·586 (ethyl).

J. L. Prosser

1798. **Analysis of certain oxychlorides of acids of phosphorus.** E. L. Gefter (Sci. Res. and Development Inst. of Plastics). *Zavod. Lab.*, 1958, **24** (6), 691.—Many acid chlorides of organic derivatives of acids of P react so rapidly with KOH that they can be determined by addition of excess of KOH and titration of the excess. Examples are—ethyl phosphorodichloridate, ethylphosphonic dichloride, 2-chloroethylphosphonic dichloride and methylphenylphosphinic chloride.

G. S. Smith

1799. **Determination of methylchloro- and phenylchloro-silanes in organic solvents with thiocyanate and ferric iron.** Toshio Takiguchi (Kiriu College of Tech., Gumma Univ.). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (5), 587–588.—Organochlorosilanes of general formula R_nSiCl_{4-n} (R=CH_3 or C_6H_5) react with KSCN or NH_4SCN in anhyd. ether to yield KCl or NH_4Cl quantitatively. An ethereal soln. of the silane (containing < 100 mg of Cl) (1 ml) is mixed with dry ether (10 ml) and $FeCl_3$ in ether (0·5%, 2 drops) and titrated with KSCN or NH_4SCN (0·1 to 0·5 *M*) in dry acetone until the soln. becomes red. The error is $< 1\%$ (relative) for methylsilanes and $< 2\%$ for phenylsilanes. This method is applicable to the determination of total Cl in mixed organochlorosilanes.

K. Saito

1800. **Aniline hydrochloride as the reaction product of organochlorosilanes and aniline. An attempt to determine some organochlorosilanes with aniline.** Toshio Takiguchi (Kiriu College of Tech., Gumma Univ.). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (10), 1236–1238.—Organochlorosilanes of a general formula R_nSiCl_{4-n} (R = CH_3, C_2H_5 or C_6H_5) react with aniline in ether to produce aniline hydrochloride $4 - n$ times the equiv. of the silane. The yield is $\simeq 97\%$ at room temp. The sample soln. in ether (0·1 to 0·5 ml) is added to anhydrous ether (water $< 0{\cdot}15\%$) containing a few drops of NH_4SCN in acetone (0·5 *M*) and $FeCl_3$ in ether (0·5%). This mixture is titrated with aniline in toluene (1 or 2 *M*) until a red colour appears. The amount of the organosilane is calculated by an empirical formula.

K. Saito

1801. **Identification and determination of polycyclic aromatic hydrocarbons in [liquid] paraffin.** H. J. Eichhoff and G. Titschack (Inorg.-Chem. Inst., Univ. Mainz). *Arzneimittel-Forsch.*, 1958, **8** (7), 376–379.—Benzopyrene, methylcholanthrene and anthracene in liquid paraffin may be identified and determined accurately in concn. of as low as 1 in 10^7 by measurement of the intensity in the appropriate spectral region of the fluorescence produced on irradiation with u.v. light.

A. G. Cooper

1802. **Quantitative determination of *p*- and *m*-chlorobenzotrifluoride by infra-red spectroscopy.** Machio Iwasaki, Hiroshige Muramatsu and Rimpei Kojima (Gov. Ind. Res. Inst., Hirate-machi, Kitaku, Nagoya). *Japan Analyst*, 1958, **7** (7), 425–429.—The *p*- and *m*-isomers of chlorobenzotrifluoride give absorption bands at 9·84 and 12·01 μ, respectively. Their spectrophotometric determination can be carried out by the use of these key bands, the working curve being linear for < 15 mg and < 30 mg of *p*- and *m*-isomer per ml of CS_2, respectively. The standard deviation is 0·4 to 0·7 % (absolute) for 10 to 80 % of *p*-isomer in an isomeric mixture, by the use of the ordinary method; by the use of the compensation or the base-line method for $< 10\%$ of *p*-isomer, the value is 0·06 to 0·10%. This method is applicable to the analysis of industrial intermediates.

K. Saito

1803. **Voltammetric determination of phenol in *iso*propylbenzene on the rotating platinum anode.** V. I. Ginzburg and T. A. Frishman (Sci. Res. Inst. of Synthetic Alcohols and Org. Products). *Zavod. Lab.*, 1958, **24** (8), 949–951.—The phenol is extracted with 4% NaOH soln. and determined polarographically in a soln. 0·17 *M* in NaOH and 0·2 *M* in boric acid.

G. S. Smith

1804. **Reactions of molybdophosphoric acid with dihydroxybenzenes. I. Colorimetric determination of catechol.** J. Strachota and Z. Kotásek (Leather Res. Inst., Otrokovice, Czechoslovakia). *Chem.*

Listy, 1958, **52** (6), 1093–1098.—The reaction of molybdophosphoric acid with catechol (**I**) and its homologues [4-methylcatechol (**II**), 3-methylcatechol (**III**)] has been used for colorimetric determination in buffered and unbuffered soln. In unbuffered soln. of pH 3·2 to 4·0 **I** can be determined when reaction conditions are strictly defined, but the reaction in phthalate buffer soln. is preferred. *Procedure*—To a Clark - Lubs buffer soln. (pH 3·80) (13 ml) in a 25-ml flask add the soln. to be determined (5 to 120 μg of **I** in 1 ml) (5 ml) and 1% molybdophosphoric acid soln. (1 ml), dilute to vol. with water and after 15 min. measure the extinction at 405 to 455 mμ. The procedure can also be used for the determination of **II** and **III** in concn. of 10 to 100 μg in 1 ml. The accuracy is ± 2%. J. ZÝKA

1805. **Identification and determination of cyclic aldehydes with 4-aminoantipyrin [4-aminophenazone].** O. Manns and S. Pfeifer (Pharm. Inst., Humboldt Univ., Berlin). *Mikrochim. Acta*, 1958, (5), 630–637.—Many cyclic aldehydes can be pptd. from fairly dil. soln. as Schiff bases with 4-aminophenazone. The crystalline product may be identified microscopically. A semi-micro method is also given for the photometric determination by means of these compounds. Photomicrographs (19) are given. D. F. PHILLIPS

1806. **Chromatography of aromatic isomers. IX. Separation of some benzene mono- and tri-carboxylic acids by paper chromatography.** J. Franc (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1958, **52** (6), 1108–1112.—The relation between dipole moment and chromatographic behaviour of *o*-, *m*- and *p*-toluic acids, hemimellitic acid, trimellitic acid and trimesic acid has been studied to work out a suitable method for their separation. The acids are converted into nitro derivatives which can be identified by their R_F values. The nitration was carried out with 0·1 g of the sample heated with 3 ml of a mixture of conc. H_2SO_4 and HNO_3 (2 : 1) at (*a*) 80° for 15 min., (*b*) at 100° for 4 hr. and (*c*) at 100% for 7 hr. The mixture was then cooled, diluted with H_2O to 5 ml, neutralised with pyridine (3 ml) and separated by ascending chromatography on Whatman No. 1 paper with the use of (*i*) *n*-butanol - pyridine - H_2O (5 : 3 : 3), and (*ii*) *iso*amyl alcohol - ethanol - pyridine - H_2O (1 : 1 : 1 : 1); $SnCl_2$ and Ehrlich's reagent were used for detection. J. ZÝKA

1807. **The detection of o-aminophenol and glyoxal by a spot reaction.** F. Feigl and D. Goldstein (Lab. da Produção Mineral, Min. da Agric., Rio de Janeiro). *Z. anal. Chem.*, 1958, **163** (1), 30–31.—Glyoxal (1 mole) reacts with *o*-aminophenol (2 moles) to form a Schiff's base which gives a red, water-insoluble complex with calcium. This reaction can be used for the detection of both glyoxal (*a*) and *o*-aminophenol (*b*), with detection limits of 1 μg and 25 μg, respectively. *Procedure*—(*a*) To a drop of test soln. add some freshly sublimed *o*-aminophenol and a small quantity of freshly ignited CaO. A red colour indicates the presence of glyoxal. (*b*) To one drop of an ethanolic or ethereal soln. of the test substance add 1 drop of 0·02% aq. glyoxal soln. and heat for 1 to 2 min. on a boiling-water bath. Cool and add a little ignited CaO. A red colour indicates the presence of *o*-aminophenol. W. T. CARTER

1808. **Study of the sulphonation reaction. XLVIII. Quantitative determination of isomeric chlorobenzenesulphonic acids.** A. A. Sprýskov and O. I. Kachurin. *Izv. Výssh. Ucheb. Zavedenií Khim. i Khim. Tekhnol.*, 1958, (1), 97–99; *Ref. Zhur., Khim.*, 1958, (21), Abstr. No. 70,639.—The method is based on the conversion of the chlorobenzenesulphonic acids into the corresponding *N*-methylanilinesulphonic acids and the conventional determination of the latter by bromination and measurement of the H_2SO_4 liberated. Convert the sulphonic acids by any known means into the sodium salts and then form the anhydrides, which are purified by vacuum-distillation (1 to 2 mm Hg). Treat ≃ 0·4 g of the mixture of anhydrides by boiling for 5 to 7 min. in a test-tube with 0·5 ml of pyridine (**I**) and 2 to 4 drops of water, add 20 to 25 mg of Cu_2Cl_2 and 2 ml of a 40% soln. of methylamine (**II**), seal the tube and heat for 17 hr. at 190° to 200°. Transfer the cooled mixture to a flask, add 1 g of Na_2CO_3 and boil to remove excess of **I** and **II**. Evaporate the soln., add 45 ml of conc. HCl and make up to 100 ml. To 50 ml of the resulting soln. in a flask with a ground glass stopper add 25 ml of 0·3 *N* bromide - bromate (**III**), set aside for 1·5 hr. at 20° to 25° and for 1 hr. at 50°, cool, add 20 ml of 0·1 *N* hydrazine hydrochloride (**IV**) and titrate excess of **IV** with 0·1 *N* **III** at 80°, with a 0·1% soln. of Tropaeolin OO as indicator. Simultaneously carry out a blank run with 5 ml of 0·3 *N* **III**, 20 ml of conc. HCl and 50 ml of water. The quantity of Br used determines the total amount of isomeric sulphonic acids. Neutralise the titrated soln. to Tropaeolin OO with aq. NH_3, extract the oil with ether, wash the extract 2 or 3 times with water (filter off solid material and wash it 3 or 4 times with water), combine the wash soln. with the main soln. and determine H_2SO_4 gravimetrically; the quantity of H_2SO_4 corresponds to the total content of the *o*- and *p*-isomers. The content of *m*-isomer is found by difference and of the *p*-isomer by the thermal method. The error in determining each isomer is < 1·5% of their total content. C. D. KOPKIN

1809. **Paper-chromatographic separation of polynuclear aromatics and their oxidation products. XXVIII. Identification of organic compounds.** J. Gasparič (Res. Inst. for Organic Syntheses, Pardubice-Rybitví, Czechoslovakia). *Mikrochim. Acta*, 1958, (5), 681–686.—The systems dimethylformamide - hexane, 1-bromonaphthalene - 90% acetic acid, and ethanol - aq. NH_3 - water (80 : 4 : 16) have proved suitable, particularly for quinones and acids. D. F. PHILLIPS

1810. **The titration of some 2-naphtholsulphonic acids with iodine.** M. Matrka and F. Navrátil (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Prûmysl*, 1958, **8** (7), 363–364.—The direct potentiometric titration of 2-naphthol-6-sulphonic acid (Schäffer acid) (**I**), 2-naphthol-7-sulphonic acid (F acid) and 2-naphthol-3 : 6-disulphonic acid (R acid) (**II**) with iodine soln. can be carried out in buffered soln. *Procedure*—Dissolve the sample (about 1 to 1·5 g) in 2·5 *N* Na_2CO_3 (3 ml) and dilute with H_2O to 250 ml. To 25 ml add 50 ml of 5% buffer soln. pH about 7 ($NaHCO_3$ - Na_2HPO_4) and titrate potentiometrically with 0·1 *N* iodine. With the use of paper chromatography, the presence of 2-naphthol-8-sulphonic acid in **I** and of 2-naphthol-6 : 8-disulphonic acid and 2-naphthol-3 : 6 : 8-trisulphonic

acid in **II** has been detected; these compounds are not substituted by iodine and do not interfere in the described procedure. J. ZÝKA

1811. **Chromatographic determination of 2-naphthylamine in 2-naphthylamine-1-sulphonic acid.** I. R. Bellobono (Ist. Tec. Ind. per Chimici, Rho, Milan, Italy). *Chim. e Ind.*, 1958, **40** (6), 457–459.—For qual. identification, various amines and aminosulphonic acids may be separated by paper chromatography and their R_F values found by using an eluent consisting of *iso*butyl alcohol - acetic acid - H_2O and detection by successive treatments with HCl, $NaNO_2$ and alkaline 2-naphthol, applied by an aerosol technique. In the determination of small amounts of 2-naphthylamine in 2-naphthylamine-1-sulphonic acid, the product is diazotised and coupled with 2-naphthol. The ppt. is extracted with benzene - *iso*butyl alcohol, the filtrate is chromatographed, and the band corresponding to the azo derivative is measured photometrically. L. A. O'NEILL

1812. **Polarographic analysis of mixtures of anthraquinones.** V. E. Ditsent. *Zavod. Lab.*, 1958, **24** (8), 951–952.—The $E_{\frac{1}{2}}$ of the polarographic waves of octahydro- and tetrahydro-2-ethylanthraquinone (products obtained during the manufacture of H_2O_2 by the 2-ethylanthraquinone method) and 2-ethylanthraquinone in a basal soln. of 0·5 *M* LiCl in 80% methanol are −0·40, −0·55 and − 0·75 V *vs.* the N.C.E. The waves are well expressed, their heights are proportional to the concn., and mixtures of the three compounds can be determined from the same polarogram. G. S. SMITH

1813. **Detection of pyridine in solution.** T. Pavolini and G. Francesconi (Padua Univ., Italy). *Chim. e Ind.*, 1958, **40** (7), 544–547.—The detection of pyridine in solution by means of methods based on iodobismuthate and cyanogen bromide reagents has been examined. The former is less sensitive but enables pyridine to be detected at a concn. of 1 in 10^4 and denatured alcohol in beverages down to 0·25%. The latter reagent, for which sulphanilic acid is preferred for the final coupling, detects 1 in 10^5 of pyridine, but alcoholic beverages tend to give a slight positive reaction on account of the presence of a compound, probably an amine, not yet identified. The composition of the reagents, the optimum working conditions and interferences have been studied. L. A. O'NEILL

1814. **The histochemical demonstration of indole derivatives by the post-coupled *p*-dimethylbenzylidene reaction.** G. G. Glenner and R. D. Lillie (N.I.A.M.D., Nat. Inst. Health, Bethesda, Md., U.S.A.). *J. Histochem. Cytochem.*, 1957, **5**, 279–296.—An historical survey is made of the chemical, biochemical and histochemical use of the benzylidene condensation reaction. The theoretical basis of the post-coupled benzylidene reaction is discussed and its specificity for tissue and use for tissues of various species is shown. A modification of the method of Lillie (*J. Histochem. Cytochem.*, 1956, **4**, 118) is described. The vitally important fixation time in formaldehyde is given new emphasis. The use of glacial acetic acid as a solvent for *p*-dimethylaminobenzaldehyde is imperative since only in this solvent can HCl produce benzylidene condensation without tissue destruction. The time of exposure is shortened to a point where the acid reaction produces the least destructive effect on collagen, yet benzylidene condensation is complete. K. R. C.

1815. **The histochemical demonstration of indole derivatives by the rosindole reaction of E. Fischer.** G. G. Glenner (N.I.A.M.D., Nat. Inst. Health, Bethesda, Md., U.S.A.). *J. Histochem. Cytochem.*, 1957, **5**, 297–303.—The benzylidene condensation of tryptophan with *p*-dimethylaminobenzaldehyde, which is dissolved in glacial acetic acid and conc. HCl, is brought about in the presence of $HClO_4$ as the condensing agent. The section is then transferred to $NaNO_2$ in a medium of glacial acetic acid and conc. HCl. The entire procedure takes only 15 min. The post-coupled *p*-dimethylaminobenzylidene reaction is only slightly more sensitive and chromogenic than the rosindole reaction, which is better for routine work. Both these reactions are superior to the xanthhydrol reaction. K. R. C.

1816. **Application of non-aqueous titrimetry to the determination of benziminazoles.** C. Hennart and E. Merlin (Orgasynthèse, Lab. de Vitry, Seine, France). *Chim. Anal.*, 1958, **40** (7), 264–265.—Sufficient of the sample to give a titre of 10 to 15 ml is dissolved in 25 ml of anhyd. propionic acid. A few drops of metanil yellow (0·2% in dioxan - anhyd. propionic acid, 1:1) are added and the mixture is titrated with 0·1 *N* $HClO_4$ (10 ml of $HClO_4$, $d = 1{\cdot}690$, dissolved in a mixture of 1 litre of anhyd. propionic acid and 35 g of propionic anhydride, and set aside for 24 hr. before use). The end-point, which is sharp and easily recognised, is a change from pale yellow to purple, and is very well placed on the potentiometric titration curve. Triplicate results for benziminazole and five alkyl-substituted derivatives show recoveries from 99·3 to 99·8%. R. E. ESSERY

1817. **Analysis of dissolved hydrocarbon gases in gasoline by combined adsorption and gas - liquid partition chromatography.** Wei-Lo Yu. *Acta Focalia Sinica*, 1957, **2** (4), 352–354.—In the method described, gases are used as the mobile phase and dibutyl phthalate on firebrick is used as column liquid for the separation of fractions below C_5. The gaseous fractions are further separated by adsorption on fuller's earth. A stream of CO_2 is passed through the column to displace the adsorbed compounds. The displaced vapour is measured in a nitrometer burette. The maximum average error in the analysis of ethane, propane, propene, butane and butene in petrol is $\gtrsim$ 0·7%. FUEL ABSTR.

1818. **Determination of halogen in gasoline: analysis of organohalogen compounds.** W. D. Garrett and J. A. Krynitsky. Washington, Off. Tech. Service, 1957, 16 pp.—A rapid method for the quantitative determination of 1:2-dichloro- and 1:2-dibromo-ethane in petrol has been developed. The organic halogen is first converted into the ionic form by the action of sodium in liquid ammonia and the resulting ionic halogen is determined by a potentiometric titration with $AgNO_3$ soln. Interfering materials are effectively removed through oxidation with $(NH_4)_2S_2O_8$. With minor modifications, the method has been applied successfully to the determination of fluorine, chlorine, bromine and iodine in several organic compounds. The accuracy compares favourably with that of established procedures. FUEL ABSTR.

1819. **Molecular sieves in gas chromatography for the determination of the normal paraffins in petroleum fractions.** B. T. Whitham (Shell Res. Ltd., Thornton, Chester, England). *Nature*, 1958, **182**, 391–392.—In the procedure described the saturated fraction of the kerosine or gas-oil is separated by solid - liquid chromatography, and a known vol. of this fraction is run through a gas-chromatographic column (200 cm × 6 mm) maintained at 164° and packed with 30 to 60-mesh "Sil-o-cel" firebrick coated with 26% (w/w) of silicone grease E 301. A pre-column (11 cm long) with 30 to 60-mesh Linde mol.-sieve 5A coated with 1·6% (w/w) of squalene is then attached to the main column and maintained at 150°. A further equal vol. of the saturated fraction of the sample is then run through the dual column to obtain the second chromatogram free from *n*-paraffins (retained by the mol. sieve). The concn. of each *n*-paraffin (C_7 to C_{18}) is calculated from the difference in corresponding peak areas between the two chromatograms when using H as carrier-gas with a thermal conductivity detector. Results agree with those obtained by gas - liquid chromatography. The mol. sieve pre-column can be used unchanged for ≃ 30 samples (≡ ≃ 85 mg of adsorbate). With a 60-cm main column at 270° to 300° and a 10-cm mol.-sieve pre-column maintained at 320° (instead of using squalene), waxes containing *n*-paraffins up to C_{40} can be analysed satisfactorily with N as carrier-gas. W. J. Baker

1820. **Rapid method for determination of aromatic contents of petroleum fractions boiling above the kerosine range.** B. M. Brook and B. T. Whitham (Shell Res. Ltd., St. Helen's Court, London). *J. Inst. Petrol.*, 1958, **44**, 212-215.—The olefin-free sample (0·5 g) is diluted with light petroleum (b.p. < 40°) and is passed through a column (50 cm × 1 cm) of freshly activated silica gel (100 to 200 mesh, ≃ 10 g). Light petroleum is added, in 10-ml amounts, until a drop of eluate (non-polar saturates) leaves no oil-stain on filter-paper. The aromatics are then eluted from the column with diethyl ether (50 ml) and weighed after removal of the ether. Reproducibility is ± 0·5%. In comparison with the standard chromatographic method, the error is within ± 0·5%, but can rise to + 2% when the content of aromatics is < 40%. A correction should be applied for oils high in S, O or N. A duplicate run takes ≃ 90 min. (45 min. if the saturates fraction is weighed and the aromatics are determined by difference). The method is applicable up to mol. wt. of 650; no dewaxing of the sample is needed.

W. J. Baker

1821. **Spectroscopic determination of traces of arsenic in petroleum naphtha and reformed spirit.** F. Wagner and R. Vitt (Scholven-Chem. A.-G., Gelsenkirchen-Buer, Dorstener Str. 227, Germany). *Erdöl u. Kohle*, 1958, **11** (7), 460–461.—Arsenic compounds in naphtha for platinum-catalytic reforming are adsorbed on Al_2O_3 by thrice shaking 1 litre of the sample with 10 mg of the chromatographic powder (30 to 40 μ). The Al_2O_3, collected by filtration, is ground in an agate mortar with 1·5 parts of a 4:1 mixture of carbon powder and KCl, and 50·0 mg of the mixture is completely volatilised from a hollow graphite electrode at 6 amp. d.c. The intensity of the resulting As line 2288·12 A is compared with standard values obtained from mixtures of benzene (600 ml) and light kerosine (400 ml) to which known amounts of triphenylarsine dissolved in *iso*propyl alcohol have been added. The accuracy is ± 20% over a range 5 to 100 parts per 10^9. A. R. Pearson

1822. **Determination of neutralisation number of lubricating greases.** DIN 51,809 and 51,812. Fachausschuss Mineralöl und Brennstoffnormung (Hamburg 1, Klosterwall 4, IV, City-Hof, Block B). *Erdöl u. Kohle*, 1958, **11** (7), 466–468.—Neutralisation no. (*n*) is defined as the acidity or alkalinity of the sample expressed in equivalent mg of KOH per g, and drafts of standard methods for determining *n* are published for criticism. In DIN 51,809, 9·15 ± 0·01 g of sample is dissolved in ethanol (40 ml ≮ 96%) containing Alkali blue 6B (1 g in 600 ml), and refluxed for 30 min. with 10 ml of 0·1 *N* aq. HCl. As substitute solvents, light petroleum or ethanol denatured by methanol, saturated with Alkali blue 6B and diluted with benzene, are permitted. The solvent (40 ml) is poured through the condenser into the cooled liquid, which is then titrated with 0·1 *N* ethanolic KOH. DIN 51,812 is for use mainly on samples too dark for colour indicators. The sample (5 ± 0·01 g) is stirred to a fine dispersion in 50 ml of diethyl ether, 20 ml of acetone and 10 ml of 0·1 *N* HCl are added, and the liquid is warmed until most of the ether and acetone is expelled. Solvent (*iso*propyl alcohol - benzene - water) (100:99:1) (130 ml) is added and the liquid is titrated potentiometrically with 0·1 *N* ethanolic KOH added in portions such that after waiting until the p.d. change is ⩾ 5 mV per min., the step is ⩾ 30 mV. Blanks are subtracted. Repeatability is ± 1 mg per g for $n \geqslant 5$, or ± 5% for $n > 5$, the corresponding reproducibility limits (different operators) being ± 2 mg per g and ± 6%. A. R. Pearson

1823. **Occurrence of oxides of nitrogen and sulphur in the determination of carbon and hydrogen in coal.** R. A. Mott and H. C. Wilkinson (British Coke Res. Assoc., London). *Fuel*, 1958, **37** (1), 62–70.—It is shown that oxides of N in the gases leaving the Liebig or Sheffield high-temp. combustion tube are negligible in amount and that the acidic gases absorbed in a MnO_2 absorber placed after the tube are mainly oxides of S. Approximately 0·013 g of these oxides can be removed by the silver spiral in the high-temp. method using "standard pushing" with the hotter end of the spiral at 860°; for higher contents of S, ≃ 1% of the sulphur oxides may pass the spiral. In the Liebig method, the oxides may pass the lead chromate packing after 40 determinations. The use of a MnO_2 absorber in both methods successfully retains the oxides of S, without impairing the efficiency. G. P. Cook

1824. **Gas - liquid chromatography. II. Analysis of the alkali extract of a low-temperature coal tar.** L. Irvine and T. J. Mitchell (Dept. of Chem. Technol., Royal Coll. of Sci. and Technol., Glasgow). *J. Appl. Chem.*, 1958, **8** (7), 425–432.—A fraction (boiling-range 230° to 250°) from the tar-acid extract of low-temp. coal tar is analysed by gas - liquid chromatography, the stationary phase being Apiezon-L stopcock grease on a Celite-535 support. Six fractions are taken, and these on further separation yield some 28 phenolic compounds, which are identified by retention-time and infra-red data from control samples. Some overlapping occurs between the various fractions, and tar, formed by thermal decomposition in the thermal-conductivity cell

appears. In some instances infra-red absorption peaks suitable for confirming the presence of a particular phenol are quoted. J. H. WATON

1825. **Analysis of low-temperature tar phenols boiling up to 234° C.** C. Karr, jun., P. M. Brown, P. A. Estep and G. L. Humphrey (U.S. Bureau of Mines, Morgantown, Va., U.S.A.). *Fuel*, 1958, **37** (2), 227–235.—Gas - liquid chromatography in combination with i.r. absorption and fractional distillation techniques was used to identify and quant. determine the phenols. Fairly good agreement was reached when comparing the results of the methods. Phenol, the three cresols, the six xylenols, the three ethylphenols, two ethylmethylphenols and two trimethylphenols were identified and determined. Retention times, other analytical data and the individual phenol concn. of a main tar distillate are listed. G. P. COOK

1826. **Methods of determining the acid, ester and iodine values of brown-coal bituminous substances.** I. A. Khrizman, V. É. Bonvech and E. M. Belzerov (Central Lab. of South Urals Geological Dept.). *Zavod. Lab.*, 1958, **24** (6), 692–694.—The sample is boiled for 30 min. with ethanol, which has been previously boiled and neutralised to thymolphthalein indicator, and then titrated hot with alcoholic 0·2 *N* KOH to give the acid value. The soln. is then boiled for 1 hr. with 25 ml of 0·2 *N* KOH (alcoholic) and the excess is titrated in the presence of thymolphthalein with 0·2 *N* acid to give the ester value. The iodine value is determined by Hubl's method. G. S. SMITH

1827. **Colorimetric absorption analysis for the identification of bitumen.** K. Letters. *Bitumen, Teere*, 1958, **9** (7), 207–208.—Bitumen solutions, 0·5%, are prepared in benzene (not in CS_2 or chlorinated hydrocarbons, in which chemical changes occur), centrifuged for 20 min. and the concentration adjusted to 0·1%. Absorption analysis in the range 4400 to 6800 A (0·25-cm cell) permits identification of the bitumen either from comparison of the full wavelength *vs.* transmission (%) curve with known curves (two examples given), or from comparison of extinction values at specific wavelengths with those for standard specimens (values given for residual and blown bitumens). The bitumens are also shown grouped according to their extinction values. J. L. PROSSER

1828. **Detection and determination of ethylenediaminetetra-acetic acid in cleansing agents and detergents.** P. Leman (Lab. Municipal d'Analyses, Lille, France). *Ann. Falsif.*, 1958, **51**, 171–172.—To 1 ml of a 10% soln. of the sample is added, in a 5-ml colorimeter tube, glacial acetic acid (0·5 ml), cobalt nitrate soln. (1%) (0·2 ml) and H_2O_2, 110 vol. (1 ml), and the resulting pink colour is measured by reference to a standard curve prepared with a 0·1% soln. of EDTA (disodium salt). A. TESSLER

1829. **The analysis of shampoos.** S. H. Newburger (Div. Cosmetics, Food & Drug Admin., Dept. of Health, Education & Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 664–668.—The usual solvent extraction procedure is unsuitable for the analysis of shampoos containing alkyl sulphate and a fatty acid - alkanolamine condensate. The alkyl suphate is therefore adsorbed from an acidified alcoholic solution on to a weakly basic resin (Amberlite CG-45, Type 2) and removed with a solution of ammonium carbonate in ammoniacal methanol. The acid eluate is evaporated and the dry residue extracted with acetone, the residue on evaporation of the acetone being weighed. The acetone-insoluble material is weighed as fatty acids. The ammoniacal eluate is evaporated to dryness and weighed as ammonium alkyl sulphate. A. A. ELDRIDGE

1830. **Analysis, in general, of essential oils by vapour - liquid partition chromatography and, in particular, of citronella and lemon-grass oils.** Y.-R. Naves and A. Odermatt. *Compt. Rend.*, 1958, **247** (7), 687–689.—Optimum conditions for rapid quant. determination of the main constituents in a sample (1 to 3 μl) of essential oil by chromatographic methods are discussed. Selection of the proper internal standard is critical. With the apparatus described previously (Naves, *Ibid.*, 1958, **246**, 2163) and a column of Reoplex 100 (1 part) supported on Celite (3 parts), operated at 180° (H or He as carrier), an analysis of citronella oil (**I**) or lemon-grass oil (**II**) can be made in $\simeq$ 20 min. With benzyl acetate (20%, w/w) as internal standard for **I**, and acetophenone (25%, w/w) for **II**, contents of citronellal, citronellol and geraniol in **I**, and of the citrals (*a* and *b*) in **II**, can be determined. The results are several per cent. lower than those obtained by the standard methods of chemical analysis (oximation, pyridine phthalisation or Burgess's method). For synthetic mixtures of the above-named constituents of each oil, the vapour partition chromatographic analyses are claimed to be accurate to within 0·5%. W. J. BAKER

1831. **Gas chromatography of terpenic hydrocarbons.** A. Liberti and G. P. Cartoni (Messina Univ., Italy). *Ric. Sci.*, 1958, **28** (6), 1192–1198.—The gas chromatography of various terpenes in an apparatus with a gas-density balance detector has been examined; retention volumes on silicone and on tritolyl phosphate stationary phases are reported. The technique has been used for the analysis of citrus oils and turpentine of various origins. Components responsible for certain bands have been identified by collection in cooled CS_2 and i.r. spectrophotometric examination. L. A. O'NEILL

1832. **Analysis of nail lacquers. III. Determination of solvent and camphor.** S. H. Newburger, M. A. Hall and J. H. Jones (Div. Cosmetics, Food & Drug Admin., Dept. of Health, Education & Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 668–673.—The solvent and camphor are isolated by vacuum-distillation. Camphor in the distillate is determined approximately by observing the ultra-violet spectrum beyond 280 mμ of a solution in ethanol. The infrared spectrum of a solution of the distillate in CS_2 is used to identify the solvents. A solution of the distillate (2·5 ml) in CCl_4 (50 ml) is dehydrated with $CuSO_4$ and used to determine its quantitative composition spectrophotometrically in the near infrared (1200 to 2400 mμ), using standard solutions of solvents and camphor. Camphor is determined separately by a procedure involving distillation, extraction, and chromatographic separation, followed by spectrophotometry at 295 mμ. Typical results are recorded. A. A. ELDRIDGE

1833. **Analysis of nail lacquers. IV. The determination of nail-lacquer solvents by gas - liquid chromatography.** J. H. Jones, C. D. Ritchie and S. H. Newburger (Div. Cosmetics, Food & Drug Admin., Dept. of Health, Education & Welfare,

Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 673–676.—A gas - liquid chromatographic procedure, in which apparatus of conventional design is used, affords a rapid and fairly accurate method for the analysis of nail-lacquer solvents. A thermal-conductivity cell is used as detector. By the use of *n*-propyl acetate as internal standard it is not necessary to know the exact sample size or to control exactly the operating variables. The most critical factor in obtaining consistent results is accurate control of the flow rate. Results for prepared solvent mixtures and for typical commercial nail lacquers are recorded.

A. A. ELDRIDGE

1834. **The paper-chromatographic identification of the amines in hair dyes.** J. Deshusses and P. Desbaumes (District Chem. Lab., Geneva). *Mitt. Lebensmitt. Hyg., Bern*, 1958, **49** (5), 335–343.—The amines used in hair dyes are identified by paper chromatography, with Whatman No. 1 paper and *n*-butanol - HCl - H_2O (7:2:4) and 2% aq. NH_3 saturated with *n*-butanol. Five different sprays make possible the identification of 13 different amines. The amines are extracted from the dye preparations and applied as their hydrochlorides. The method described has been used to examine 14 dyes of Swiss, German or French origin. All but one contained *p*-tolylenediamine. The other components found with varying frequency were *m*-tolylenediamine, *m*-phenylenediamine, *m*- and *p*-aminophenols and aminodiphenylamine.

S. BAAR

1835. **Micro-determination of dyes by amperometric titration. II. Titration of 2:6-dinitrophenolindophenol with ascorbic acid.** Tadahiko Ogawa (Fac. of Engng, Yokohama Univ., Minamiku). *J. Electrochem. Soc. Japan*, 1958, **26** (7), 323–326.—Indication of the end-point for the redox titration of 2:6-dinitrophenolindophenol (**I**) with ascorbic acid (**II**) is best effected amperometrically (− 0·85 V *vs.* the S.C.E.) in the presence of metaphosphoric and acetic acids (pH 2). **I** (0·08 to 5·0 mg) is dissolved in an acid mixture (3 g of metaphosphoric acid and 8 g of glacial acetic acid in 50 ml of water) (5 ml), diluted to 10 ml and titrated with **II** soln. (0·1 to 1 mg per ml) with a dropping mercury electrode (≃ 50 drops per sec.) at room temp. The error is ≃ 2% and the time taken for an estimation is < 40 min.

III. Titration of chrysoidine and safranine with tungstosilicic acid. Tadahiko Ogawa. *Ibid.*, 1958, **26** (9), 406–409.—Chrysoidine (**III**) and safranine (**IV**) are amperometrically titrated with tungstosilicic acid at − 0·40 V *vs.* the S.C.E. in 4 *N* H_2SO_4 (10 ml) and at − 0·60 V in 8 *N* H_2SO_4 (10 ml), respectively. The pptn. proceeds swiftly at room temp. in a molar ratio 1 (reagent) to 4 (dye). The error for 0·2 to 8 mg of **III** and 0·7 to 7 mg of **IV** is 1·5% and the time for estimation is ≃ 30 min.

K. SAITO

1836. **Determination of heavy metals in dyes for foodstuffs.** Z. Malkus (Inst. of Hyg., Prague). *Průmysl Potravin*, 1958, **9** (7), 362–363.—The polarographic method for the determination of Cu and Zn and the colorimetric method for the determination of Pb and Fe have been found suitable for the determination of impurities in dyes for foodstuffs. *Procedure for Zn and Cu*—Ignite the sample (10 g) in a porcelain dish and heat in an electric furnace. Dissolve the cooled residue in a mixture (1:1) of HNO_3 (1:1) with HCl (2:1) (1 ml), transfer with *N* aq. NH_3 to a 25-ml flask, dilute to 15 ml, separate the ppt. by centrifuging, transfer the supernatant liquid (10 ml) to a polarographic vessel, add a saturated soln. of Na_2SO_3 and gelatin soln. (0·5%) and register the polarographic waves of Cu and Zn. Evaluate the results with the method of standard additions. *Procedure for Pb and Fe*—Ignite and heat the sample as in the previous example, dissolve the residue in a mixture of HNO_3 and HCl (5:1) (1 ml) and dilute to vol. in a 25-ml flask. Determine Pb in 20 ml of this soln. by an indirect colorimetric method, with Cu diethydithiocarbamate (Šedivec and Vašák, *Chem. Listy*, 1952, **46**, 607) and Fe in 5 ml of the remaining soln. with 1:10-phenanthroline. By using this method, 1·5 to 14·5 mg of Cu, 4·0 to 140 mg of Zn, 0·1 to 4·1 mg of Pb, and 120 to 680 mg of Fe, per litre, were determined.

J. ZÝKA

1837. **Estimation of the active substance in dressing baths of mineral oil basis.** G. Grajetzki and J. Tutschek (VEB Thüring Artificial Fibre Works "Wilhelm Peck" Schwarza). *Faserforsch. u. Textiltech.*, 1958, **9** (8), 338–339.—In a rapid (20 min.) works-method for assessing the oil content of dressing baths (for staple fibre) of mineral oil basis, the sample is heated with conc. HCl to break the oil emulsion, then centrifuged to effect quant. separation of the oil, the vol. of separated oil serving to fix the fat content. Graphs are given to show the oil content/fat content relationships.

H. L. WHITEHEAD

1838. **Cleanliness of fillings and stuffings for bedding, upholstery, toys and other domestic articles.** British Standards Institution (2 Park St., London). B.S. 1425:1954, Amendment No. 7.—A limit of ≯ 1·5% of soluble extracted matter when tested by the prescribed method is prescribed for kapok and mixtures of akund or cotton with kapok whether loose or layered.

N. E.

1839. **Quality of fillings for bedding upholstery, etc. Part 2. Curled hair and curled hair/fibre mixtures loose or layered.** British Standards Institution (2 Park St., London). B.S. 2894: Part 2:1958, 18 pp.—Definitions are given and processes of manufacture are specified for new and re-processed curled hair and hair - fibre mixtures. The size of sample and manner of sampling are laid down, and standards are specified for cleanliness and composition. Apparatus and procedures are described for determining cleanliness, by weighing the material extracted at 20° by shaking for 20 min. with water - 0·880 aq. NH_3 - methanol - acetone (41·5:1·2:7·5:10); for animal fibre content, by weighing the cellulosic residue after boiling with 5% NaOH soln.; and for N by the Kjeldahl method.

R. E. ESSERY

1840. **Gas-chromatographic analysis of impurities in commercial methyl acrylate.** Yuzi Takayama (Mitsubishi Rayon Co., Kyobashi, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (6), 682–684.—Methyl propionate (**I**) (< 10%) and ethyl acrylate (**II**) (< 10%) in commercial methyl acrylate are determined by gas chromatography by the use of Carbowax 400 as fixed phase and He as carrier (rate of flow, 42 ml per min.). The peaks are satisfactorily separated at 83·5° and both **I** and **II** are determined with methyl acetate as internal standard. The error is < 1% and the limit of determination ≃ 0·05%. Water vapour can be determined by the use of tritolyl phosphate as fixed phase.

K. SAITO

1841. **Determination of methacrylic acid in the intermediates for the production of methyl methacrylate.** Tsugio Takeuchi, Motohisa Furusawa and Yuzi Takayama (Fac. of Engng, Yamanashi Univ., Motoyanagi-cho, Kofu). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (5), 561–563.—For the determination of methacrylic acid (**I**) produced by the hydrolysis of methacrylamide (**II**), **I** is steam-distilled in the presence of tartaric acid (to prevent the hydrolysis of **II**) and Na_2SO_4 (to increase the rate of distillation of **I**) and titrated with NaOH soln. No interference results from $(NH_4)_2SO_4$, methyl methacrylate and a small amount (< 0·2 g) of 2-hydroxy*iso*butyric acid. Acetone cyanohydrin (**III**) vitiates the estimation. This method is useful for the analysis of the intermediates at the later stages of the hydrolysis of **III**. *Procedure*—Dissolve the sample containing < 200 mg of **I** in water (100 ml) or in the smallest vol. of aq. methanol (for the intermediates of the esterification process). Neutralise with 0·5 *N* NaOH to methyl red, add tartaric acid (5%, 10 ml), a small amount of zinc and Na_2SO_4 (35 g) and dilute to 200 ml. Distil and titrate the distillate (120 ml) with 0·1 *N* NaOH to phenolphthalein. K. Saito

1842. **The paper-chromatographic detection of ε-caprolactam in the presence of ε-aminocaproic acid [6-aminohexanoic acid].** K. Czerepko (Med. Akad., Bialystok, Poland). *Mikrochim. Acta*, 1958, (5), 638–645.—The best separation of the two compounds is from water - alcohol soln., *e.g.*, butanol - propanol - H_2O (2:2:1). Potassium bismuth iodide used for the identification of caprolactam will detect as little as 10 μg. D. F. Phillips

1843. **Use of polarography for studying certain plastics.** V. D. Bezuglyĭ and V. N. Dmitrieva (Kharkov Dental Materials Works). *Zavod. Lab.*, 1958, **24** (8), 941–947.—The polarographic determination of methyl, ethyl, butyl and allyl methacrylates, vinyl cyanide, dibutyl phthalate, phenyl salicylate, benzoyl peroxide and quinol is studied. To determine methyl methacrylate and dibutyl phthalate in the polymer, the sample (1 g) is dissolved in 10 ml of benzene and the soln. is diluted to 50 ml with methanol. The clear soln. (1 ml) is mixed with 5 to 10 ml of a saturated soln. of tetramethylammonium iodide in 92% methanol, and the polarographic wave with $E_{\frac{1}{2}} = -1{\cdot}88$ V, which is composed of that due to methyl methacrylate and the second wave of dibutyl phthalate, is measured. A further sample (1 to 2 g) is treated with 20 ml of benzene and set aside for several hours. A portion (1 ml) of the clear soln. is mixed with 9 ml of the same basal soln. as before and the first wave of dibutyl phthalate ($E_{\frac{1}{2}} = -1{\cdot}77$ V) is measured. Contents are calculated from calibration curves. To determine benzoyl peroxide in emulsion-polymerised methyl methacrylate, the sample (1·5 to 1·8 g) is dissolved in 20 ml of benzene and the soln. is treated with methanol to precipitate the polymer. The soln. is treated with 2 g of NH_4NO_3 and 1 ml of 0·2% methyl red soln. and then diluted to 100 ml with methanol. The polarogram is taken over the range + 0·45 to − 0·2 V ($E_{\frac{1}{2}} = +0{\cdot}27$ V) and the content is found from a calibration curve or by the method of addition. G. S. Smith

1844. **Determination of polyhydric alcohols in paint media.** C. P. A. Kappelmeier and J. Mostert (Kunsthars. Synthese N.V., Katwijk-aan-Zee, Holland). *Peint.-Pigm.-Vern.*, 1958, **34** (7), 309–310.—Alkyd resins are subjected to aminolysis with 2-phenylethylamine. On addition of $CHCl_3$, pentaerythritol and trimethylolethane crystallise out, while trimethylolpropane and glycerol remain in solution. The analysis of certain mixtures with the aid of this separation is described. L. A. O'Neill

1845. **Polarographic determination of free sulphur in rubber and rubber-compounding chemicals.** O. Rozací (Res. Inst. of Rubber and Plastics Technol., Gottwaldov, Czechoslovakia). *Chem. Prûmysl*, 1958, **8** (7), 385–387.—The method of Proske (*Kautsch. u. Gummi*, 1940, **16**, 13) for the polarographic determination of free sulphur in the acetone extract of rubber has been modified by choosing a more suitable medium. *Procedure*—Dissolve the acetone extract prepared from 1 g of the rubber sample or from 0·2 to 1 g of the rubber-compounding chemicals in 5 to 10 ml of pyridine and add 25 to 50 ml of the electrolyte [0·3 *M* ammonium acetate in glacial acetic acid - methanol (1:1)]. Remove O and register the wave from 0·2 to − 2·8 V. Compare with a calibration curve. The addition of 2 or 3 drops of a 1% soln. of gelatin in glacial acetic acid to the soln. of ammonium acetate is recommended. Large amounts of tetraethylthiuram disulphide interfere; other sulphur-containing compounds commonly present in the samples do not influence the determination. J. Zýka

1846. **The determination of pentaerythritol tetranitrate in the presence of *cyclo*trimethylenetrinitramine.** J. Staněk and J. Vacek. *Chem. Prûmysl*, 1958, **8** (7), 361–363.—Selective reduction with $FeCl_3$ and the determination of the Fe^{3+} formed with $TiCl_3$ soln. is the basis of the determination of pentaerythritol tetranitrate (Pentrite) (**I**) in admixture with *cyclo*trimethylenetrinitramine (hexahydro-1:3:5-trinitro-*s*-triazine; Hexogen) (**II**). *Procedure*—Into a 500-ml flask place the sample containing ≯ 0·22 g of **I** and 0·12 g of **II**. Dissolve in glacial acetic acid (30 ml) by heating and add 25% HCl (25 ml). Remove air with a stream of CO_2, add 0·7 *N* $FeCl_3$ (50 ml) and heat for 20 min. under reflux. After 10 to 15 min. the soln. becomes clear and the colour changes from deep green to bright orange. After 10 min. cool, add 10% NH_4SCN soln. (10 ml) and titrate with 0·2 *N* $TiCl_3$ till colourless. Add 20 ml of *N* $TiCl_3$ and 30% sodium citrate soln. (25 ml) and heat again for 30 min. under reflux. Cool and titrate with 0·25 *N* $(NH_4)_2SO_4.Fe_2(SO_4)_3$ until the red colour remains unchanged for 1 min. Carry out a blank. The whole titration must be carried out in a stream of CO_2. The mean error is ± 0·3%. J. Zýka

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See also Abstracts—**1605**, Determination of formaldehyde. **1606**, Use of BrCl in determination of phenols and deriv. **1608**, Use of ICl in determination of org. comp. **1735**, Determination of EDTA. **1860**, Partition chromatography of organic acids. **1863**, Chromatographic identification of phenol derivatives. **1864**, Chromatography and electrophoresis of phenols. **1895**, Determination of thujone. **1896**, Determination of (−)piperitone. **1926**, Testing of plastics for use with foods. **1945**, Determination of hydrocarbons in air. **1959**, Determination of diphenylamine. **1978**, Separation of hydrocarbons by continuous gas-elution chromatography. **1981**, Determination of hydrocarbons and oxygenated deriv. by gas chromatography.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

1847. **Multiple colorimetric indicator for rapid determination of the pH of biological liquids.** J. F. Frattini (Lab. Bioquím., Dirección General de Sanídad del Ejército Argentino, Argentina). *Rev. Asoc. Bioquím. Argentina*, 1958, **23**, 142–147.—The indicator is prepared from dimethylamino-azobenzene (0·3 g), bromothymol blue (0·2 g), phenolphthalein (0·5 g), ethanol (80 ml) and H_2O (100 ml). It shows a distinct colour change for each unit of pH from 2 to 10 (2 red, 3 salmon, 4 orange, 5 yellow, 6 green - yellow, 7 green, 8 dark green, 9 blue - green, 10 violet). For examining the acidity of stomach contents, the initial pH of the filtered sample (1 or 10 ml) is determined, and followed by titration with 0·01 *N* or 0·1 *N* NaOH, first to pH 3·5 (free HCl), then to pH 6 (org. acids and acid salts), and finally to pH 8·5 (chloro-organic acids). L. A. O'NEILL

1848. **An improved histochemical method for the demonstration of potassium.** J. R. Crout and R. B. Jennings (Northwest. Univ. Med. School, Chicago). *J. Histochem. Cytochem.*, 1957, **5**, 170–177.—Pptn. is produced by sodium cobaltinitrite soln. Freeze-dried tissue and absolute avoidance of water before staining are the modifications employed. The use of $(NH_4)_2S$ after staining is not recommended. Spectrophotometric extinctions at 768 mμ of digested tissues show that K is completely pptd. by this technique and that there is no loss or shift of K during the staining process. K. R. C.

1849. **Radiochemical determination of caesium-137 in foodstuffs, human urine and other biological samples.** Noboru Yamagata, Toshiko Yamagata, Shunji Matsuda, Eisaku Tajima and Sadakata Watanabe (Kiriu College of Tech., Gumma Univ., Kiriu). *Japan Analyst*, 1958, **7** (7), 433–438.—Caesium-137 in fall-out is collected together with other alkalis by the HCl - ethanol method and separated from them by the chloroplatinate pptn. method. Both ^{40}K and ^{87}Rb are pptd. together, but the amount of the former is < 2 disintegrations per min. (corresponding to 1 mg of K) and the soft β-rays of the latter do not affect the γ-counting of ^{137}Cs. Grain (2 to 3 kg) and meat (3 to 4 kg) are charred and the black powder is extracted with HCl (1 + 1, 300 ml). Vegetables (1 kg) and milk powder (500 g) are ashed and similarly extracted with HCl. Human urine and blood (1 litre) are decomposed with HNO_3, charred and extracted with HCl. Soil (3 kg) is extracted with *M* ammonium acetate, evaporated and decomposed with aqua regia. The HCl extract is mixed with CsCl (carrier, $\simeq$ 25 mg), treated successively with $BaCl_2$, aq. NH_3 soln., ammonium carbonate and ammonium oxalate, evaporated to dryness and the Cs separated as described above. K. SAITO

1850. **Radiochemical determination of strontium-89 and strontium-90 in vegetation, fish and seaweed ash.** U.K.A.E.A. Chemical Services Dept. (Operations Branch, Windscale). U.K.A.E.A., Report IGO-AM/W-185, 1959, 14 pp.—Strontium carrier (50 mg of Sr) is added to the ash and the mixture is fumed with HNO_3 and $HClO_4$. After the pptn. of the Sr and any Ca as phosphates the Sr is separated by repeated pptn. of the nitrate. Ferric hydroxide and $BaCrO_4$ scavenges are followed by pptn. of $SrCO_3$, which is β-counted. The $SrCO_3$ is dissolved and ^{90}Y is separated from the soln. after a period of at least 10 days. Full details are given of the counting procedures and the standardisation of the counting equipment. G. J. HUNTER

1851. **Micro-determination of thorium. V. Determination of a microgram amount of thorium in plants, shells and coral.** Shinnosuke Higashi (School of Gen. Educ., Kyoto Univ., Sakyo-ku, Kyoto). *Japan Analyst*, 1958, **7** (7), 441–444.—Thorium in these samples is pptd. with Fe, Ca and PO_4^{3-}, the ppt. is ignited, fused with borax (3 g) and extracted with water (total 100 ml) or HCl. The soln. is made pH 4·5 with 0·1 *N* NaOH to precipitate Fe, Th and PO_4^{3-}, the ppt. is filtered off and treated with 3 *N* NaOH (30 ml) to precipitate Fe and Th. Thorium is then extracted with *cyclo*-hexanone (20 ml) from an HNO_3 soln. (15 ml) of pH 0·65 containing 10 g of NH_4NO_3 and determined photometrically (600 mμ) with *o*-arsonophenyl-azochromotropic acid (neothorin) (Ishibashi and Higashi, *Anal. Abstr.*, 1955, **2**, 2387) at pH 1·0, the small amount of Fe remaining being reduced with hydroxylamine hydrochloride soln. (1%, 2 ml). Plants are charred and ashed at 450° to 600°, a portion ($\simeq$ 5 g) is extracted with 3 *N* HCl (50 ml) and neutralised with aq. NH_3. The ppt. is treated with HF, HNO_3 and $HClO_4$, heated and fused with borax. Shell or coral (50 to 100 g) is ignited, dissolved in HCl and neutralised, with Fe ($\simeq$ 10 mg) added as carrier, and treated similarly. K. SAITO

1852. **Semi-micro technique for iron-59 determinations in biological systems, using beta-counting in a liquid scintillator.** T. P. Leffingwell, G. S. Melville, jun., and R. W. Riess. *U.S. Atomic Energy Comm.*, Rep. AF-SAM-58-93, 1958, 6 pp.—After acid digestion of Fe-containing components of biological systems, the Fe may be selectively complexed and extracted from the digestant soln. The coloured complex is of adequate stability to permit spectrophotometric measurement of the Fe present. If ^{59}Fe is present in the system, the same coloured complex may be introduced into a liquid scintillator soln. and the beta emanations counted with an efficiency of $\simeq$ 50%. The technique offers an approx. 25-fold increase in effective counting as compared with the commonly used gamma-counting technique, and up to a 15-fold reduction in initial sample size. NUCL. SCI. ABSTR.

1853. **Separation and determination of milli-microgram amounts of cobalt and microgram amounts of copper in biological material.** A. Smoczkiewiczowa and W. Mizgalski (Balneoclimatic Inst., Poznan). *Nature*, 1958, **182**, 53–54.—Cobalt and copper are separated from other metals present in blood by paper chromatography. The blood sample is dried and ashed, and the ash is dissolved in acids. The soln. is chromatographed on Whatman No. 1 filter-paper rolled into the form of a cylinder with the sample streaked 3 cm from the lower end which dips into conc. aq. NH_3. After the solvent front has proceeded about 10 to 12 cm above the starting line, the paper is removed, inverted and dried at 80°. The paper strip is then ashed carefully on a piece of silver foil and the ash analysed by emission spectrography, with the foil as one electrode and a silver rod at 7 mm distance as the other. The wavelengths of the spectrum range

from 2500 A to 7000 A and a photograph is obtained by exposing a G22 plate with 150 diaphragm breadth for 10 sec. Standard curves are obtained by treating soln. of known composition in the same way. The quantities of Co and Cu are determined by measuring the intensities of the 3453·5 A and the 3247·5 A lines, respectively. Results are tabulated and compared with those given by other methods.
S. M. MARSH

1854. **Micro-determination of glucose by titrating with zinc sulphate the ferrocyanide produced by the reduction of alkaline ferricyanide solution.** J. A. C. van Pinxteren (Pharm. Lab., Rijksuniv., Utrecht, Holland). *Pharm. Weekbl.*, 1958, **93** (17), 753–795.—The oxidising soln. is composed of 0·005 *M* $K_3Fe(CN)_6$ mixed with an equal vol. of a soln. containing 2·24% of KOH and 34·8% of K_2HPO_4 immediately before use. This soln. (5 ml) is standardised by heating for 10 min. in boiling water with a slight excess of glucose, adding 1 ml of 4 *N* H_2SO_4 and 2 drops of Fe^{3+} soln. and titrating potentiometrically to the dead-stop end-point with 0·0075 *M* $ZnSO_4$. Blood or serum may be deproteinised by tungstate before the titration. A table is given showing the amounts of glucose (50 to 300 μg) equivalent to ml of 0·0075 *M* $ZnSO_4$. The results are in good agreement with those obtained by the Hagedorn-Jensen method.
P. S. ARUP

1855. **Estimation of benzene-extractable bilirubin (modification of Rivoire's method).** R. Besson and R. Fauvert (Hôpital Beaujon, Clichy). *Rev. Franç. Étud. Clin. Biol.*, 1958, **3** (6), 673–674.—A modification of the method of Rivoire (*Ann. Méd.*, 1946, **47**, 197) is described, in which two extractions with benzene are used to recover the extractable portions of bilirubin from urine, serum or bile. Bilirubin is measured in the extracts by photometry (blue filter, or at 420 mμ) and comparison with a standard curve. If only one extraction is made, the recovery of extractable bilirubin is about 75%. Normal serum contains 5 to 8 mg per litre of benzene-extractable bilirubin; values to be expected in various types of jaundice are given.
D. W. Moss

1856. **A semi-micro method for the determination of crystal violet sorbed to biological material.** R. Fischer (Univ. Hosp., Saskatoon, Canada). *J. Histochem. Cytochem.*, 1958, **6** (4), 237–243.—The amount of crystal violet and iodine taken up by bacteria, wool, nerve- and muscle-fibres and certain proteins is determined quant. After iodination and subsequent washing, the bacterial suspension is spotted on filter-paper. Wool fibres are wound on to the paper. Continuous extraction of the dye is carried out by a procedure similar to one-dimensional ascending chromatography, with the organic phase of Partridge's *n*-butanol-acetic acid-water solvent (4:1:5). The dry paper is extracted with boiling ethanol (95%) until it is colourless. The extinction of the extract is measured at 591 mμ. The parts played by "loosely bound" iodine and covalently bound iodine in the crystal violet uptake of intact and alkali-degraded wool are described. The crystal violet uptake before and after iodination is compared, and the theoretical implications are discussed. K. R. C.

1857. **Measurement of absorption and fluorescence spectra of paper chromatograms. Determination of specific activity of carbon-14-labelled compounds on chromatography paper.** F. Korte and H. Weitkamp (Univ. Bonn, Germany). *Angew. Chem.*, 1958, **70** (14), 434–437.—Absorption and fluorescence spectra obtained from chromatograms with an Ulbricht sphere, a reflecting device contained, in this case, in the standard Beckman reflectance unit, have been measured on a recording spectrophotometer. The absence of wasteful elution stages enables the activity of carbon-14-labelled compounds on the same paper to be determined independently. Provided that the chromatograms are run to provide spots less than 0·9 cm in diameter, which will fit into the optics, the concn. concerned can be calculated directly from the absorption obtained, using the Kubelka-Munk relationship—

$$[(1 - R)^2/2R]K \equiv \text{const.},$$

where $R \equiv \log I_0/I$ and K is concn. in μg per ml. A range of between 0·1 and 5 μg can be conveniently handled. A small change in $\lambda_{max.}$ between reflection spectra and the normal transmission spectra of eluted solutions is attributed to the energy of elution.
E. G. CUMMINS

1858. **Separation of carbohydrates on coconut-charcoal columns.** F. W. Barth and T. E. Timell (McGill Univ., Montreal, Canada). *Canad. J. Chem.*, 1958, **36** (9), 1321–1322.—Uronic acids, methylated monosaccharides and other mixtures of sugars can be rapidly and completely separated on columns (12 cm × 4 cm) of pretreated coconut-charcoal (50 to 200 mesh). The main fractions of the sample are eluted with aq. ethanol, *e.g.*, 4, 7 and 15%, respectively, for a mixture of uronic acids from the part hydrolysis of milkweed floss. Neutral sugars should be eluted with 1·5% aq. ethanol; 4-O-methyl-D-glucose can be eluted with 5% aq. ethanol. The charcoal should be pretreated successively with 6 *N* HCl, H_2O, anhyd. ethanol and finally H_2O.
W. J. BAKER

1859. **Spectrophotometric determination of total carbohydrate [in algae].** B. R. Hewitt (Dept. of Agric. and Stock, Toowoomba, Queensland). *Nature*, 1958, **182**, 246–247.—The carbohydrate content of plant material is determined by a modification of the method described by Fales (*J. Biol. Chem.*, 1951, **193**, 113) and Schlegel (*Planta*, 1956, **47**, 510). An algal suspension (diluted × 5) (1 ml) is added to 10 ml of anthrone reagent (0·2 g of anthrone, 8 ml of ethanol, 30 ml of water and 100 ml of conc. H_2SO_4) and heated for 7 min. on a boiling-water bath. The extinction of the coloured product is measured at 620 mμ. Beer's law is maintained only when the suspension to reagent ratio is low (1:10). The error for duplicate samples is $< 8\%$.
S. BAAR

1860. **Partition chromatography of organic acids: variations in solvent composition.** R. P. Harpur (Inst. of Parasitology, McGill Univ., Macdonald College P.O., Que., Canada). *Canad. J. Biochem. Physiol.*, 1958, **36** (7), 707–715.—To investigate the effect of polar solvent concn. in the elution of organic acids from a column of silica which has been ground with 0·06 *N* H_2SO_4, various mixtures of $CHCl_3$, water and *n*-pentanol are used and the "peak volume" (volume of eluent at which the max. amount of acid is eluted) and the spread of the elution peaks are determined for acetic, pyruvic, fumaric, lactic, succinic, α-oxoglutaric, *cis*-aconitic, oxalic, malic, citric and tartaric acids. It is shown that a plot of log "peak vol." against log percentage *n*-pentanol is linear for each acid, and that the spread of an elution peak is proportional to its "peak vol."

The results are applied to the planning of a stepwise elution of various acids. Succinic and α-oxoglutaric acids are not separated completely, and oxalic acid tails badly. E. J. H. BIRCH

1861. **Sensitive micro-spectrophotometric method for succinate analysis.** M. B. Waitzman and E. Q. Adams (Western Reserve Univ. Sch. of Med., Cleveland, Ohio, U.S.A.). *Nature*, 1958, **182**, 129.—In the assay of succinate by reduction of a redox dye in a succinic dehydrogenase and cyanide-poisoned system, re-oxidation of the dye by atmospheric oxygen is avoided by displacing air by argon in the soln. and in the stoppered Beckman cells. The cells are placed in a jar in an argon atmosphere, and charged with 0·1 ml of a 20% succinoxidase suspension, 0·3 ml of a 0·1 *M* phosphate buffer (pH 7·4), 0·1 ml of 0·05 *M* KCN, 0·5 ml of 0·01% dichloroindophenol soln., and 1·8 ml of glass-distilled water. Reduction by endogenous substrate is complete in 6 min., at which point 0·2 ml of water is added to the blank, and 0·2 ml of succinate soln. to all other cells. The decrease in extinction is measured at 620 mμ. Each 0·01 Beckman density unit is proportional to 0·00167 μmole of succinate. An accuracy within 0·01 μmole can be achieved. R. E. ESSERY

1862. **Analytical determination of (−)-phenylacetylcarbinol.** O. Bauerová and Š. Bauer (Dept. Pharm. Chem. and Biochem., Chem. Inst. Acad. Sci., Bratislava, Czechoslovakia). *Chem. Zvesti*, 1958, **12** (6), 366–369.—A paper-chromatographic method for the determination of (−)-phenylacetylcarbinol in crude "ketol" has been worked out. The system *iso*amyl alcohol - water with ascending chromatography and detection with blue tetrazolium have been used. In the range 10×10^{-6} to 70×10^{-6} g the amount of (−)-phenylacetylcarbinol corresponds to the size of the spots, when evaluated planimetrically, after photometric measurement. The mean error is ± 5%. J. ZÝKA

1863. **Method for the paper-chromatographic separation and identification of phenol derivatives, mould metabolites and related compounds of biochemical interest, using a "reference system."** L. Reio (Wenner-Gren Inst., Dept. of Physiol. Chem., Univ. of Stockholm, Sweden). *J. Chromatography*, 1958, **1** (4), 338–373.—The procedure involves the preparation of six chromatograms with six specified solvent systems. The material is loaded on to each paper (Whatman No. 1, 24 cm × 55 cm) in a thin transverse line 20 cm long, with a reference spot of some known compound at each end of the line, the ascending technique being used. After drying, each paper is examined in u.v. light, and then sprayed in narrow (10 mm) longitudinal bands, with each of ten specified reagents, by using an air-brush fed with air, N or CO_2 at 2·4 atm. pressure. The observed colours are numbered according to a specified series of 72 standard tints prepared by means of the "Derwent" series of coloured pencils. The R_F values for each of the six solvents, and the observed colours with u.v. light and each of the ten spray reagents, are recorded for known substances on punched cards, to form the reference system mentioned, to which the results obtained from test material are referred. An illuminated cabinet, fitted with two air-brushes, is described and illustrated, in which the six chromatograms can be conveniently sprayed and examined, and the preparation of the solvents, spray reagents, and standard tints is described. R_F values for the six solvents, and colour reactions with u.v. light and with the ten spray reagents, are tabulated for some 450 compounds, and the data for the various groups of substances, the preparation and use of the reference system, and the application of the procedure to the preliminary investigation of unknown mixtures, are discussed. R. E. ESSERY

1864. **Paper chromatography and paper electrophoresis of phenols and glycosides.** C. B. Coulson and W. C. Evans (Dept. of Agric. Chem., Univ. Coll. of North Wales, Bangor). *J. Chromatography*, 1958, **1** (4), 374–379.—Two new solvent systems are given for the chromatography of phenols, aglycones, glycosides and certain sugars, with the ascending technique on Whatman No. 4 paper at 20° ± 1°. (*i*) Benzene - glacial acetic acid - water (4:1: just saturated) gave results in 8 hr.; (*ii*) *n*-butanol - ethanol - borate buffer (9·54 g per litre of sodium tetraborate) (1:1:1), with a paper impregnated with the buffer and dried before use, gave results in 18 hr. Spots were detected by (*a*) a spray based on the *o*-dihydric phenol colour reaction of Evans (*Biochem. J.*, 1947, **41**, 376), 10% sodium molybdate soln. - 0·5 *N* HCl (2:1) being sprayed first, followed by 0·5 *N* $NaNO_2$ and then by 0·5 *N* NaOH; this gives a strong pink with catechols; (*b*) diazotised *p*-nitroaniline, followed by Na_2CO_3 soln.; this gives a cherry-red to purple colour, except when borate buffer has been used, when the colours are yellowish brown. R_F values and colour reactions for these solvents and sprays are listed for 43 substances. Horizontal paper electrophoresis on Whatman No. 4 paper, for 7 hr. at 10 V per cm and 5 to 7 mA, with borate buffer, was also useful, and M.G.D. values (the ratio of the migration of the substance to the migration of DNP-glycine), and colours given with the diazo spray reagent, are tabulated for 40 substances. Data are also given for a number of sugars and related compounds, alkaline $AgNO_3$ soln. being used for detection, and for various glycosides and aglycones, with u.v. light for detection. R. E. ESSERY

1865. **The fluorimetric measurement of deoxyribonucleic acid in animal tissues with special reference to the central nervous system.** J. M. Kissane and E. Robins (Dept. of Pharmacol., Washington Univ. Sch. of Med., St. Louis, Mo., U.S.A.). *J. Biol. Chem.*, 1958, **233** (1), 184–188.—The method is based on the measurement of the fluorescence produced in the reaction between 3:5-diaminobenzoic acid dihydrochloride and deoxyribose (liberated from the deoxyribonucleic acid by the strongly acidic dihydrochloride). The method, which is specific for deoxyribonucleic acid, permits measurement of as little as 2·4 mμg in a final volume of 50 μl, and does not measure interfering substances which give high results in the diphenylamine reaction. The results obtained agree with those given by u.v. absorption methods. J. N. ASHLEY

1866. **Analysis of mixtures of glucosamine and galactosamine with bakers' yeast.** B. M. Pogell and D. F. Koenig (Wilmer Ophthalm. Inst., Johns Hopkins Univ., Baltimore, Md., U.S.A.). *Nature*, 1958, **182**, 127–128.—Bakers' yeast ferments D-glucosamine completely, but does not utilise D-galactosamine. Tissue hydrolysates are adsorbed on and eluted from Dowex-50 resin (H^+ form) to isolate the amino sugars from other carbohydrates,

and the total hexosamine is determined in an aliquot by the Blix - Elson - Morgan method (*cf. Brit. Abstr. C*, 1949, 292; Boas, *Anal. Abstr.*, 1954, **1**, 340). A second aliquot (up to 1 mole of glucosamine) is added to 1·5 ml of water, treated with 2 ml of yeast suspension (600 mg of wet yeast in phosphate buffer, pH 7·5), and the flask is shaken at 70 oscillations per min. at 30° for 1 hr. After removal of yeast by centrifuging at 4°, aliquots (1 ml) are analysed for hexosamine. A galactosamine and a glucosamine standard, and a yeast control, are included. Galactosamine is calculated, using the galactosamine standard, from the hexosamine remaining after yeast action, and glucosamine is obtained by difference from total hexosamine. Results for known mixtures and for blood-group substances are satisfactory. The reproducibility of the method is equal to that of the Blix test. R. E. ESSERY

1867. **Polarographic behaviour of α-lipoic acid.** J. Sponar and M. Jirsa (Charles' Univ., Prague, Czechoslovakia). *Biochem. Biophys. Acta*, 1958, **29** (2), 434–435.—α-Lipoic acid may be determined from the total reduction wave in a phosphate buffer at pH 6·8. Calibration is rectilinear up to concn. of 10^{-3} *M*. Interpretation of the polarograms in relation to those for cysteine is discussed. W. H. C. SHAW

1868. **The use of iodine for the detection of lipids.** M. W. Whitehouse, A. E. Bresler and E. Staple (Biochem. Dept., School of Med., Univ. of Pennsylvania, Philadelphia, U.S.A.). *J. Chromatography*, 1958, **1** (4), 385–386.—The chromatogram or electrophoretic strip is thoroughly dried and exposed overnight in a closed chamber containing iodine crystals, lipids being revealed as brown or yellow zones. Alternatively, the dried paper may be dipped in 0·2% iodine soln. in light petroleum or diethyl ether, and the excess of iodine is removed by exposure to a gentle stream of air. Examination under u.v. light facilitates the detection of weakly staining compounds. Semi-quant. data are tabulated for the sensitivities of 32 compounds. R. E. ESSERY

1869. **Amino-acid analysis by horizontal paper chromatography.** J. Chartier, F. W. van Klaveren and G. Vaillancourt (Fisheries Res. Board of Canada, Grande-Rivière, P.Q., Canada). *J. Chromatography*, 1958, **1** (4), 317–326.—The development of a quant. method for the separation of amino acids in protein hydrolysates by circular paper chromatography is discussed, with illustrations of suitable equipment. A preliminary separation into 6 zones is achieved on a Whatman No. 1 paper, 34 cm diam., in which three slits have been cut with a double razor, producing two segments with apical angles of 30°. The material is loaded at the apex of each segment, and developed with *n*-butanol - 2-butanone - water - aq. NH_3 (5:3:1:1), the zones being located on one segment with ninhydrin, and the appropriate portions of the second segment being cut out. From these, the acids are transferred, by elution in a specified manner, on to Whatman No. 1 K.C.T. paper (Kawerau, *Chromatog. Methods*, 1956, **1**, 7), 26·5 cm diam., which is prepared with 5 equally-spaced radial slits. Zones 1 (aspartic and glutamic acids), 3 (histidine, alanine, serine), and 6 (leucine, *iso*leucine, phenylalanine) are transferred to different segments of one paper, which is developed with *tert.*-butyl alcohol - formic acid - water (75 : 0·8 : 24·2), while zones 2 (lysine, arginine, glycine) and 4 (valine, methionine, tyrosine) are transferred to different segments of a second paper, which is developed with lutidine - water (2:1). Zone 5 contains tyrosine only. R. E. ESSERY

1870. **Chromatography of amino acids on alumina "plates."** M. Mottier (Eidg. Gesundheitsamt, Bern, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1958, **49** (6), 454–471.—Chromatography on alumina "plates" (thin beds of adsorbent spread on glass plates) has been successfully applied to 22 amino acids and a protein hydrolysate. The method of preparing the "plates" is described in detail. The quality and degree of activation of the alumina are important, and best results were obtained using material from Merck (No. 1097) activated for 0·75 hr. at 300° to 500°. In order to obtain quant. displacement, the starting point must be made alkaline with *N* NaOH. The most suitable developing solvent is *n*-butanol - ethanol - H_2O (7:3:3 for use in summer, and 3:2:2 for use in winter), and the developing process should be carried out slowly at room temp. The spots are indicated by treatment with 0·2% soln. of ninhydrin in ethanol containing 20% by vol. of glacial acetic acid. *o*-Coumaric acid may also be used as an indicator since the medium is alkaline. The chromatograms are fixed with paraffin wax. The addition of surface-active agents does not give significantly better results. Advantages of the method are its rapidity (6 to 8 hr. at room temp.) and the possibility of using very small amounts of substance (0·5 to 1·5 μg or 0·001 *M* soln. of amino acid). The method may be applied to the control of the purity of individual amino acids, the analysis of hydrolysates and in biological chemistry. S. M. MARSH

1871. **The use of turbidity measurements in the microbiological determinations of amino acids. II. Arginine, cystine, histidine and tyrosine.** S. S. Schiaffino, J. J. McGuire and H. W. Loy (Food & Drug Admin., Dept. of Health, Education & Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 679–681.—Turbidity determinations (*cf. Anal. Abstr.*, 1959, **6**, 1031) were made after incubation at 34° ± 0·5° for 18 hr. Standard reference curves are given for the response to *Streptococcus faecalis* of arginine and histidine, and for that to *Leuconostoc mesenteroides* of cystine, histidine and tyrosine. Satisfactory comparative results are recorded. A. A. ELDRIDGE

1872. **Absolute colorimetric procedure for the simultaneous determination of O-DNP-tyrosine and ε-N-DNP-lysine.** E. R. Fritze and H. Zahn (Chem. Inst., Univ., Heidelberg, Germany). *Z. anal. Chem.*, 1958, **162** (6), 414–423.—O-DNP-tyrosine (**I**) and ε-N-DNP-lysine (**II**) are determined in the presence of each other by photometric measurement at 300 mμ and 360 mμ of a dil. HCl soln. containing from 3×10^{-5} to 1×10^{-4} mole per litre. The concn. of **I** and **II** are evaluated with the help of their molar absorption coeff. at these wavelengths. When the procedure is applied to the analysis of protein hydrolysates, humic substances are removed by passing the soln. through a small column of Filter-Cel. Good agreement with theoretical amounts is shown for silk, but for wool the content of **I** is 4% high, owing to the adsorption of 2:4-dinitrophenol on to the threads. Moreover, wool appears to have more reactive groups than correspond to tyrosine alone. J. H. WATON

1873. **Nitration and determination of phenylalanine.** D. Monnier and R. Guerne (Lab. de Chim. Minérale et de Chim. Anal., Univ. de Genève, Switzerland). *Anal. Chim. Acta*, 1958, **19** (1), 90–100 (in French).—The nitration products (including some nitrobenzoic acids) formed by heating phenylalanine with various concn. of HNO_3 for various periods of time were studied by polarographic and chromatographic methods. To determine phenylalanine, a weighed sample is heated under reflux in a tube (10 cm × 2 cm) with 0·5 ml of 10 *N* HNO_3 for 90 min., with a cold finger condenser. The cooled nitration mixture is neutralised with KOH soln. to methyl red, buffer soln. (pH 6) is added and after passage of hydrogen for 10 min. the soln. is polarographed. At concn. of $2{\cdot}4 \times 10^{-4}$ *M* and 6×10^{-3} *M*, the error is 2·5%, but at concn. of the order of 2×10^{-5} *M* it is about 12%. The determination can be carried out on 30 μg in 10 ml of soln. F. J. M.

1874. **A tryptophan histochemical method.** N. C. Brummer, M. J. Carver and L. E. Thomas (Univ. Missouri, Columbia, Mo.). *J. Histochem. Cytochem.*, 1957, **5**, 140–144.—This method is based on the use of nitrous acid followed by a soln. of N-1-naphthylethylenediamine dihydrochloride (2%) in *N* HCl, the exposure time in each case being 15 min. The nitrous acid is prepared by mixing equal vol. of $NaNO_2$ soln. (8%) and HCl (6 *N*). Tests proving the specificity of the reaction are described. Results of measurements of the i.r. absorption of the coloured reaction-product, using free tryptophan and other similar compounds, are given. The structure of the reaction product is postulated. K. R. C.

1875. **A spectrophotometric method for the determination of reduced glutathione.** Y. Avi-Dor and R. Lipkin (Israel Inst. for Biol. Res., Ness Ziona). *J. Biol. Chem.*, 1958, **233** (1), 69–72.—The method is based on that of Avi-Dor and Mager (*cf. Anal. Abstr.*, 1957, **4**, 999). Addition of borate to the product of interaction between fluoropyruvic acid and thiol compounds which have no free amino group in the α- or β-position causes a shift in the absorption band from 275 to 290 mμ, with a corresponding increase in absorbancy. The high extinction of the resulting borate complex is utilised for the determination of reduced glutathione. The method can be used to determine a mixture of cysteine and reduced glutathione. J. N. Ashley

1876. **Chromatographic detection of minute quantities of thyroxine in serum.** V. Štolc (Slovak Acad. Sci., Bratislava, Czechoslovakia). *Nature*, 1958, **182**, 52.—Modification of a procedure described by Bowden *et al.* (*Biochem. J.*, 1955, **59**, 93) has given a method which will detect thyroxine after chromatography of only 1 ml of serum. The chromatogram is sprayed with 0·2 *N* ceric ammonium sulphate and *N* sodium arsenite (1 : 1) and left for 15 min. to dry at room temp. after superficial desiccation. It is then passed through a 0·5% soln. of fluorescein in acetone or a 5·0% soln. of anthranilic acid in acetone. After drying the chromatogram in a current of air, thyroxine is detected by viewing in u.v. light. Alternatively, the chromatogram can be sprayed with 1% *o*-toluidine soln. in acetone when pale-yellow spots appear on the dark-brown background indicating the position of thyroxine. The colour is stable for a considerable time. The sensitivity of the method is about 0·01 μg of thyroxine. S. M. Marsh

1877. **The quantitative determination of iodoamino acids of thyroid tissue.** T. H. Kennedy (Univ. of Otago, Dunedin, N.Z.). *Austr. J. Biol. Sci.*, 1958, **11** (1), 106–113.—The ability of three different pancreatic preparations to liberate iodoamino acids from thyroid tissue is studied. A hydrolysis is described which produces less destruction of organic iodine-containing compounds, with reduced formation of iodide. A thyroid homogenate (100 mg of tissue) is made 2 *N* with respect to NaOH by addition of the reagent (10 *N*). After the addition of thiourea (100 μg) the contents of the tube are thoroughly de-gassed. The sealed tubes are heated at 98° to 100° for 16 to 18 hr. The excess of Na^+ is removed and the hydrolysate is run through a column of cation-exchange resin in the acid form. The column is then washed with aq. NH_3 soln. (1·0 *N*) until the residual ^{131}I is less than 1% of the total. Most hydrolysates were also analysed by paper chromatography. The results are tabulated and good reproducibility indicates that both methods are satisfactory. K. R. C.

1878. **Zone electrophoresis in a density gradient. Stability conditions and separation of serum proteins.** H. Svensson, L. Hagdahl and K.-D. Lerner (LKB-Produkter Fabriksaktiebolag, Stockholm). *Sci. Tools*, 1957, **4** (1), 1–10.—The stability conditions in zonal-density-gradient-electrophoresis are studied mathematically. Various ways of securing permanent stability are suggested. The maximum possible mass-content of a migrating zone is calculated and is shown to increase with the square of the zone thickness. Successful separations of human serum are described, from which it is concluded that the resolving power of the zonal-density-gradient method is comparable to that of ordinary electrophoretic procedures. The regular appearance of a pre-albumin fraction is stressed. K. R. C.

1879. **Quantitative evaluation of paper electropherograms of serum proteins.** J. Pieper (Physiol.-Chem. Inst., Univ. Münster, Westf.). *Klin. Wochschr.*, 1958, **36** (13), 605–609.—Replicate strips of paper-electrophoretic separations of serum proteins were evaluated and compared by several methods. These were extinction measurements with filtered light, followed by planimetry (Elphor); automatic determination with monochromatic light (Elphor Integraph), extinction measurements with automatically recorded curve followed by planimetry (Zeiss extinction recorder II), dye elution with 0·05 *N* NaOH followed by spectrophotometry, and a method similar to that of the Integraph but using a non-transparent strip (Spinco). All the methods were compared with that of dye elution and all recording methods tended to give low albumin values. This error is decreased if monochromatic light is used. Automatic determination with the Integraph increased the over-all error of the estimations. The necessity for the periodic checking of planimeters is stressed and a moderately sensitive automatically recording instrument followed by planimetric evaluation of the curves is recommended. S. Baar

1880. **The determination of protein-bound iodine.** G. A. Robinson and H. A. DeLuca (Dept. of Biochem., Univ. of Western Ontario, London, Canada). *Canad. J. Biochem. Physiol.*, 1958, **36** (7), 701–706.—The method of Barker *et al.* (*J. Clin. Invest.*, 1951, **30**, 55) is modified to give greater speed. *Procedure*—To 0·5 ml of plasma (heparinised or

oxalated, but not citrated) are added 0·5 ml of 10% $ZnSO_4.7H_2O$ soln. and 0·5 ml of 2% NaOH soln. The ppt. is centrifuged and washed and, after the addition of 0·5 ml of 20% Na_2CO_3 soln., it is dried for 10 to 15 min. in a quick drying oven consisting of a number of copper tubes which can hold the test-tubes, heated over a hot-plate under jets of air directed downwards to prevent bubbling. The dried ground protein is ashed at 640° ± 10° for 1·5 to 2 hr. and dissolved in 0·5 ml of 4 *N* HCl. After effervescence ceases, 2 ml of 2 *N* H_2SO_4 is added and the tube is centrifuged for 5 min. To 0·5 ml of 1% sodium arsenite soln. is added 2 ml of the acid supernatant liquid. The samples are kept at 39° and 1·0 ml of 2% ceric ammonium sulphate soln. is added at the same temperature. The extinction is measured at 425 mμ after exactly 10 min. The standard soln. are prepared by addition of NaI to ashed egg-albumin precipitates (which contain no iodine). Standards in which protein has not been ashed give a different result as some interference with the iodine catalysis of the arsenite - ceric reaction occurs. The recovery of iodine is 95·0 to 96·5%. E. J. H. Birch

1881. **Spectrophotometric determination of protein [in algae] in alkaline solution.** B. R. Hewitt (Dept. of Agric. and Stock, Toowoomba, Queensland). *Nature*, 1958, **182**, 246.—A modified Folin phenol method for the determination of total algal protein is described. The reagents used are (*A*) 1 ml of 0·5% $CuSO_4.5H_2O$ soln. in 1% K tartrate soln., mixed with 50 ml of 2% Na_2CO_3 soln. in 0·1 *N* NaOH, and (*B*) the Folin - Ciocalteu reagent diluted 1:1 with water. An algal suspension (5 ml) is centrifuged, and the washed cells are extracted with *N* NaOH on a boiling-water bath. After centrifuging, the extract is added to 5 ml of water, then 1 ml of this dilute soln. is added to 5 ml of reagent *A* and allowed to stand for 10 min. Reagent *B* (0·5 ml) is added and the extinction is read after 40 min. at 750 mμ. With duplicate samples the error was within 5%. S. Baar

1882. **Characterisation of yeast proteins by paper chromatography.** H. Vögeli (Inst. f. Haustierernährung, ETH Zürich). *Mitt. Lebensmitt. Hyg., Bern*, 1958, **49** (6), 471–477.—The amino acids present in a protein hydrolysate obtained from yeast have been separated and characterised by two-dimensional paper chromatography. Most of the acids were separated by the descending phase method, with butanol - glacial acetic acid - water as solvent in the first run and phenol - water in the second run. The remaining acids were separated by using ascending phase chromatography with butanol - ethyl methyl ketone - water and butanol - methanol - water as solvents. Twenty-two amino acids were definitely identified and two unknown substances isolated, one of which was probably homoserine. Correlation of the chemical composition and biological value of the protein is discussed, but it is concluded that, as yet, such correlation can only be approximate.

S. M. Marsh

1883. **Paper-chromatographic methods for the rapid quantitative determinations of steroids.** K. Albrecht and G. Wix (Res. Inst. for the Pharm. Ind., Budapest). *Magyar Kém. Foly.*, 1958, **64** (7–8), 237–240.—A new method is described for the paper-chromatographic separation of progesterone, 11α-hydroxyprogesterone and 6β:11α-dihydroxyprogesterone. After placing the drop at the bottom of the paper strip, the part of the strip above the drop is impregnated with propane-1:2-diol (**I**) - methanol (3:7) (this procedure is used in all subsequent examples) and the chromatogram is run with toluene - **I** as solvent. Photometric determination in abs. ethanol at 242 mμ has an accuracy of ± 5% if 75 to 150 μg of steroid is present; otherwise it is ± 10%. In this, and the following separations, the ascending method on S. & S. 2045 (or b) or Macherey-Nagel 214 paper was generally used. Two other, new, solvent systems are also recommended; the R_F values obtained are shown in brackets. Methylcyclohexane - CCl_4 (1:1), saturated with **I**, is recommended for the separation of progesterone (0·86), androst-4-en-3:17-dione (0·68), androsta-1:4-diene-3:17-dione (0·41) and Δ^1-dehydrotestololactone (0·03). Toluene - dichloroethane (1:1), saturated with **I**, is used for the separation of Reichstein's compound S (0·51), cortisone (0·21) and cortisol (0·07). The determination of this group was carried out by the methods of Mader and Buck, or of Porter and Silber, or by 2:2′-dipyridyl; photometric determination at 242 mμ can also be used. In all these, the accuracy is ± 10%.

A. G. Peto

1884. **Determination of 17-oxosteroids in urine. II. Comparative investigation of the distribution of 17-oxosteroids in the urine of normal women and those suffering from cancer by the use of various hydrolysis conditions.** W. Staib and W. Schild (Physiol. Chem. Inst., Med. Acad., Düsseldorf, Germany). *Klin. Wochschr.*, 1958, **36** (13), 600–604.—Acid and enzymatic hydrolyses are used in the determination of the 17-oxosteroid fractions, particularly 11-hydroxy-5β-androstanolone in the urine of normal and carcinoma patients. *Procedures*—(*a*) A 48-hr. sample of urine is refluxed for 30 min. with 0·1 vol. of 50% H_2SO_4. The hydrolysate is extracted with ether for 48 hr., washed once with 1/3 vol. of 10% NaOH and twice with 1/3 vol. of H_2O, dried with Na_2SO_4 and evaporated *in vacuo* below 40°. (*b*) A 48-hr. sample of urine is adjusted to pH 4·5 with 0·1 vol. of *M* Na acetate buffer; 50 to 100 ml of $CHCl_3$ is added and the specimen is incubated for 72 hr. at 37° in the presence of 300 i.u. of glucuronidase. Then 0·1 vol. of 50% H_2SO_4 is added and the steroids are extracted with ether for 48 hr. The ether is separated and the remaining urine is refluxed for 30 min. and twice extracted with 2 vol. of ether. The pooled ether extracts are concentrated to ≃ 200 ml and washed and dried as described above. The dried residue is dissolved in 10 ml of benzene. Gradient elution chromatography, paper chromatography and i.r. analysis were carried out as described earlier (*cf. Klin. Wochschr.*, 1958, **36**, 166). Carcinoma patients showed a decreased excretion of dehydro*epi*androsterone and patients with early mammary cancer showed increased excretion of 11-hydroxy-5β-androstanolone.

S. Baar

See also Abstracts—**1652**, Determination of B in vegetable matter. **1782**, Determination of fructose. **1783**, Removal of electrolytes from sugar soln. **1789**, Ion-exchange chromatography of organic acids. **1814**, **1815**, Histochemical demonstration of indole derivatives. **1891**, Determination of quinic acid in tobacco. **1970**, Sample feeder for electrophoresis.

Pharmaceutical analysis

1885. **Titration of some organic bases with perchloric acid.** M. Rink, R. Lux and E. Franken (Pharm. Inst., Univ. Bonn, Germany). *Dtsch. ApothZtg*, 1958, **98** (26), 660–663.—Amidopyrine, nicotinic acid, nikethamide, isoniazid, nicotinamide, procaine hydrochloride, Larocaine hydrochloride, choline chloride, methylamphetamine hydrochloride, codeine hydrochloride, lobeline hydrochloride, thiamine hydrochloride, dihydromorphinone bitartrate and dihydrocodeinone bitartrate have been titrated with $HClO_4$ in anhydrous soln. The indicator Fettblau gave the best results. M. H. SAWISTOWSKA

1886. **A study of the titration of organic bases in chlorobenzene with acetous perchloric acid.** M. Pernarowski and D. W. Blackburn (School of Pharmacy, Purdue Univ., West Lafayette, Ind.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (8), 585–589.—Potentiometric titrations can be carried out in chlorobenzene with glass and sleeve-type calomel electrodes; 0·05 *N* $HClO_4$ in glacial acetic acid is the most suitable titrant. Galvanometer response is rapid but not erratic. The optimum concn. of titrant at the end-point is approx. 1 vol. of titrant for every 5 vol. of soln. Bases as weak as caffeine can be determined in chlorobenzene as solvent, but only with limited accuracy. A mixed solvent containing 20% of glacial acetic acid in chlorobenzene can be used for the titration of organic bases insol. in chlorobenzene. Bromophenol blue is a suitable indicator for titrations in chlorobenzene to a visual end-point. The results of 6 potentiometric titrations of atropine showed an average recovery of 99·7% and a standard deviation of 0·55%. A. R. ROGERS

1887. **Separation and identification of major alkaloids from opium by partition chromatography on paper.** G. Nadeau, G. Sobolewski, L. Fiset and C. G. Farmilo (Biochem. Dept., Hôpital St. Michel Archange, Quebec, Canada). *J. Chromatography*, 1958, **1** (4), 327–337.—Previous work on the separation of opium alkaloids is discussed, and a method developed for the two-dimensional chromatography of acetic acid extracts of opium without further purification. For the first operation, the material is spotted on to Whatman No. 1 paper, which has been pretreated with 2% aq. $(NH_4)_2SO_4$ soln. and dried, and developed by ascending chromatography in a specified manner with *iso*-butyl alcohol - acetic acid (5:1, saturated with water by a specified method), this taking 16 to 17 hr. according to temperature and size of chamber. The chromatogram is examined under u.v. light (cotarnine, narcotine and papaverine fluoresce) and stained with potassium iodoplatinate soln. (morphine, deep blue, other alkaloids, shades of purple). This procedure does not separate narcotine and papaverine, and, if this is required, the chromatogram, before staining, is given a second treatment, at right angles to the first, with water-saturated diethyl ether, after allowing a pre-saturation period of 3 hr. in the chromatography chamber before adding the mobile organic phase. The process is complete in 2 hr. R_F values and "codeine ratios" (the ratio of the distance travelled by the alkaloid to that travelled by a reference spot of codeine) are tabulated for the first solvent, and R_F values for the second dimension for morphine, codeine, thebaine, papaverine and narcotine. These values were rather variable, but the narcotine to papaverine ratio remained reasonably constant between 1·13 and 1·21, mean 1·16. Results are given for opium extracts. R. E. ESSERY

1888. **The quantitative separation of papaverine from narcotine in mixtures.** L. Kum-Tatt and C. G. Farmilo (Food and Drug Directorate, Dept. of Nat. Health and Welfare, Ottawa). *J. Pharm. Pharmacol.*, 1958, **10** (7), 427–435.—In the presence of excess of $CHCl_3$, papaverine (**I**) but not narcotine (**II**) forms an insoluble reineckate in acid soln. This forms the basis of a method of quant. separation of **I** and **II** (*cf. Anal. Abstr.*, 1958, **5**, 2755), which is applied to the determination of **I** and **II** in opium. *Procedure*—Triturate the sample (4·5 g) with glacial acetic acid (25 ml) followed by H_2O (20 ml) and filter. Extract a 10-ml aliquot with $CHCl_3$ (8 × 10 ml) and wash each extract in turn with H_2O (15 ml), H_2O (15 ml) plus NaOH soln. (1:1) containing a little $NaHSO_3$ (15 ml), H_2O (15 ml), 0·1 *N* H_2SO_4 (15 ml and 10 ml) and 0·05% $NaHCO_3$ soln. (10 ml). Evaporate the combined washed extracts to dryness on a water bath. Dissolve the residue in CCl_4 (50 ml), strain through cotton-wool and pass through a column of $Ca(OH)_2$. Wash the column with CCl_4 (2 × 10 ml) and extract the combined CCl_4 fractions with 0·1 *N* HCl (2 × 10 ml). Shake the HCl soln. with $CHCl_3$ (10 ml) for 10 min., add 2% ammonium reineckate soln. (10 ml), shake for 30 min. and filter through sintered glass. To determine **I**, dissolve the ppt. in acetone and measure the extinction at 525 mμ. To determine **II**, shake the $CHCl_3$ layer of the filtrate with 0·25% $AgNO_3$ soln. (40 ml), separate and further extract with $CHCl_3$ (2 × 10 ml); strain the combined $CHCl_3$ fractions through cotton-wool, dilute to 250 ml, and either measure the extinction at 310 mμ or evaporate and titrate with 0·05 *N* $HClO_4$ in glacial acetic acid. A. R. ROGERS

1889. **Determination of reserpine in tablets by infra-red spectrophotometry.** W. R. Maynard, jun. (Virginia Dept. Agric., Div. Chem. & Foods, Richmond, Va., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 676–679.—Reserpine, deserpidine and rescinnamine have similar infra-red spectra, but have differences sufficient for specific distinction. A $CHCl_3$ solution is used. The total alkaloids are determined (against reserpine standards) at 5·78 μ, and rescinnamine at 7·58 μ. Total extinction at 6·1 μ gives a measure of reserpine and rescinnamine; the contribution of the latter to absorbance at 6·1 μ is calculated and the balance attributed to reserpine, since deserpidine does not absorb at that wavelength. The deserpidine content is calculated from the total absorbance at 5·78 μ less that due at that wavelength to the other two alkaloids. A. A. ELDRIDGE

1890. **A note on the determination of caffeine in tablets.** C. M. P. Wirth (Sidney Ross Co., S. A., Admon. de Correos 51, Mexico). *Drug Standards*, 1958, **26** (3), 85.—The value of the method of Wirth (*Drug Standards*, 1952, **20**, 226) for the determination of caffeine in the presence of aspirin and phenacetin is confirmed. A. R. ROGERS

1891. **Micro-colorimetric method for the determination of quinic acid and its content in flue-cured tobacco.** M. Nagasawa (Tobacco Res. Dept., Cent. Res. Inst., Japan Monopoly Corp.). *Bull. Agric. Chem. Soc. Japan*, 1958, **22** (4), 205–207 (in

English).—The periodate oxidation product of quinic acid (**I**) reacts with nitroprusside and piperazine to give a yellow colour which is measured photometrically. To 1 ml of test soln. (25 to 120 μg of **I**) add 1 ml of aq. sodium periodate soln. (0·64%, fresh daily), and maintain at 30° for 15 min. Then add 1 ml of ethanediol soln. (30 ml dissolved in 100 ml of water), shake, and hold at 30° for 5 min. Remove from the bath, add 1 ml of carbonate buffer (pH 10·3) (1 part of 0·1 *M* $NaHCO_3$ with 9 parts of 0·05 *M* Na_2CO_3) and 1 ml of nitroprusside - piperazine soln. freshly prepared (0·5% each of sodium nitroprusside and piperazine in water, diluted 10 times). Mix well, heat at 100° for 5 min., cool for 5 min. in running water, make up to a convenient vol. (usually 10 ml) with 95% ethanol, read the extinction in a 1-cm cell at 390 mμ against a blank. Refer the results to a calibration curve prepared from known amounts of **I** similarly treated at the same time. The method has been applied to methanolic tobacco extracts, after descending chromatography on Whatman No. 1 paper, with amyl alcohol - 5 *N* formic acid (1:1). Flue-cured tobacco contains 0·23% of **I** (dry).

R. E. Essery

1892. **Colorimetric estimation of digitalis glycosides with 2:4-dinitrophenyl phenyl sulphone.** D. H. E. Tattje (Pharmacog. Lab., State Univ. of Groningen, Holland). *J. Pharm. Pharmacol.*, 1958, **10** (8), 493–498.—The method described previously (*cf. Anal. Abstr.*, 1958, **5**, 4277) has been studied in detail. Higher extinction coeff. are obtained if the glycosides or aglycones are boiled with 0·4 *N* HCl before the examination. A. R. Rogers

1893. **Separation of some hydroxyanthraquinones by filter-paper electrophoresis.** A. C. Core and E. R. Kirch (Coll. of Pharm., Univ. of Ill., Chicago). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (7), 513–515.—Separation of 14 different hydroxyanthraquinones has been accomplished by the hanging-strip technique of paper electrophoresis. Best results were obtained with a barbiturate buffer of pH 8·6 and ionic strength 0·05 on Whatman No. 3MM paper at a const. potential of 400 V for 16 hr. To prepare extracts of aloes, cascara or senna for chemical determination, remove the inactive, free hydroxyanthraquinones by extraction with $CHCl_3$, then extract with aq. ethanol, hydrolyse the extracted glycosides and oxidise anthrones to hydroxyanthraquinones with H_2O_2.

A. R. Rogers

1894. **Vegetable purgatives containing anthracene derivatives. VIII. The paper chromatography of certain anthraquinones and their glycosides.** T. J. Betts, J. W. Fairbairn and V. K. Mital (School of Pharm., Univ. of London). *J. Pharm. Pharmacol.*, 1958, **10** (7), 436–441.—Values of R_F determined for chrysophanol, aloe-emodin, aloe-emodin anthranol, emodin and rhein by ascending paper chromatography with toluene as solvent are given; Whatman No. 20 paper is preferred. For the separation of anthraquinone glycosides, develop by the ascending technique on Whatman No. 1 or 3 MM paper with the lower phase of a mixture of water - acetone - benzene (2:1:4). To locate the compounds, dry the chromatograms, spray with 0·5% magnesium acetate in methanol, heat at 90° for a few minutes and examine by u.v. light. A. R. Rogers

1895. **Colorimetric determination of thujone with 3:5-dinitrobenzoic acid.** D. H. E. Tattje (Lab. voor Farmacog. en Galen. Farmacie, Rijksuniv., Groningen, Holland). *Pharm. Weekbl.*, 1958, **93** (15), 689–694.—To 4 ml of an ethanolic soln. of thujone add 5 ml of a 4% w/v soln. of 3:5-dinitrobenzoic acid in ethanol and 3 ml of 4 *N* NaOH, and measure the colour intensity in a 0·5-cm cell at 537·5 mμ against a freshly prepared control of 4 ml of ethanol, 5 ml of reagent and 3 ml of 4 *N* NaOH, the reaction being carried out at 20°. The max. colour is attained in 12 min. for α-thujone and 3 min. for β-thujone, after the addition of the NaOH. The calibration curve is rectilinear, E (1%, 1 cm) being 72·0 for α-thujone and 104·2 for β-thujone. The reagent and the soln. can be stored for 3 days without deterioration. By this method cedar leaf and tansy oils give results in good agreement with those obtained by the hydroxylamine method. Oils of thuja and of sage give much lower results by the colorimetric method. With wormwood oil the β-thujone content found is high. P. Haas

1896. **Colorimetric determination of (−)-piperitone with 3:5-dinitrobenzoic acid.** D. H. E. Tattje (Lab. voor Farmacog. en Galen. Farmacie, Rijksuniv., Groningen, Holland). *Pharm. Weekbl.*, 1958, **93** (15), 694–700.—Mix 4 ml of a soln. of (−) - piperitone (up to 1·6 mg) in ethanol with 5 ml of a 4% w/v soln. of 3:5-dinitrobenzoic acid in ethanol and add 2 ml of 3 *N* NaOH. After about 40 min. measure the colour intensity in a 0·5-cm cell at 537·5 mμ with a slit width of 40 A against a control of 4 ml of ethanol, 5 ml of reagent and 2 ml of 3 *N* NaOH, the reaction being carried out at 20°; a fresh control should be used for each determination. The calibration graph between 0 and 1·6 mg is slightly curved. The value of E (1%, 1 cm) diminishes from 135·6 [0·2 mg of (−)-piperitone] to 121·4 [1·6 mg of (−)-piperitone]. Cineole does not react with the reagent. The value of E for (−)-α-phellandrene is 2·3 and consequently the influence of this substance is insignificant. P. Haas

1897. **A rapid assay method for penicillin.** R. P. Patel and P. B. Chemburkar (Dept. of Pharm. Technol., L.M. College of Pharmacy, Ahmedabad). *Indian J. Pharm.*, 1958, **20** (6), 159–163.—By use of *Bacillus megatherium* in the cup-plate assay of penicillin, incubation for 4 or 4·5 hr. at 37° is sufficient. The diameter of the zone of inhibition is a linear function of the logarithm of the concn. of penicillin in the range 1 to 30 units per ml. A turbidimetric method also gives satisfactory results after incubation for 3 hr. A. R. Rogers

1898. **Non-aqueous titration of the tetracycline antibiotics and some commercial preparations.** F. Yokoyama and L. G. Chatten (Dept. of Nat. Health and Welfare, Ottawa, Canada). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (8), 548–553.—To assay the pure antibiotics, dissolve the sample (50 mg) in nitromethane (50 ml) and formic acid (1 ml) with stirring, add benzene (5 ml) and titrate with 0·05 *N* $HClO_4$ in dioxan either potentiometrically with silver - silver chloride and glass electrodes, or visually to a green end-point with a mixed indicator (0·1% of methylene blue and 0·2% of quinaldine red in anhydrous methanol) (0·1 ml). When the sample is a hydrochloride salt, add 6% mercuric acetate in glacial acetic acid (1 ml) before the titration. The method may be applied with slight modifications to the assay of tablets, capsules

or suppositories. When the tablets contain magnesium stearate as lubricant, titrate to a blue colour before the addition of mercuric acetate. With ointments, increase the volumes of benzene and formic acid and titrate the soln. while hot. Recovery experiments show close agreement with microbiological and spectrophotometric procedures. The coefficient of variation is $\simeq +0{\cdot}5\%$.

A. R. ROGERS

1899. **Determination of insulin by paper chromatography.** J. Bouman and J. D. H. Homan (Res. Lab. N.Y. Organon, Oss, The Netherlands). *Biochim. Biophys. Acta*, 1958, **29** (2), 417–422.—Chromatography is carried out on circular Whatman No. 20 papers (30 cm diameter) developed with the upper phase of a mixture of *sec.*-butyl alcohol and 1% (v/v) acetic acid (1:1) for 15 hr. After drying, the paper is treated with a soln. of bromophenol blue, $HgCl_2$ and acetic acid for 15 min. Excess of dye is washed out with aq. acetic acid (3%), the paper is air-dried, treated with ammonia vapour and the spots corresponding to insulin (R_F 0·3 to 0·4) from standards and test soln. are cut out. After elution with 0·01 *N* KOH, the extinctions are measured at 595 mμ and the potency of the sample is calculated by the method given. Results obtained were found to agree with biological results and to be of similar precision for materials of potency > 19 i.u. per mg. For samples of lower potency, standards of similar purity are required. W. H. C. SHAW

1900. **Spectrophotometric determination of steroid esters.** A. A. Forist and S. Theal (Res. Lab., The Upjohn Co., Kalamazoo, Mich.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (7), 520–522.—Mix an approx. 0·005 *M* soln. of the sample in ethanol (5 ml) with a filtered, freshly prepared mixture of 12·5% hydroxylamine hydrochloride in methanol - 12·5% NaOH in 85% methanol (1:1) (3 ml). Allow to react at room temp. for 10 min. (cortisol acetate or prednisolone acetate), or 30 min. (cortisol *cyclo*pentylpropionate or prednisolone *cyclo*pentylpropionate), or 40 min. (17α-acetoxyprogesterone), or at 50° for 60 min. (testosterone propionate). Add ethanolic ferric perchlorate reagent (30 ml) and leave in the dark for about 10 min. Dilute with the reagent to 50 ml, mix and measure the extinction in a 1-cm cell at 530 mμ against a reagent blank. Simultaneously analyse a standard soln. of the steroid ester and calculate by Beer's law. The mean recovery is 99·9% with a coeff. of variation of 1·4%, estimated from the results of 25 determinations. A. R. ROGERS

1901. **Determination of testosterone *cyclo*pentylpropionate and testosterone present in oily solutions, by countercurrent separation.** M. Vitali, P. del Grande and G. Pancrazio (Lab. Ricerche ORMA-ITR, Rome). *Ann. Chim., Roma*, 1958, **48** (8–9), 622–629.—Countercurrent separation is used for determination of testosterone and testosterone *cyclo*pentylpropionate present together in sesame-oil soln., with successive identification and spectrophotometric determination. Satisfactory results are obtained with a 10-stage apparatus with a special flask (illustrated). The following extinction coeff. in ethanol are used—testosterone, 565 at 240 mμ, and testosterone *cyclo*pentylpropionate, 409 at 240 mμ. C. A. FINCH

1902. **Polarographic determination of prednisone in tablets.** H. P. Deys and J. A. C. van Pinxteren (Pharm. Lab., Rijksuniv., Utrecht, Holland). *Pharm. Weekbl.*, 1958, **93** (17), 760–765.—A self-registering apparatus with dropping mercury and calomel electrodes is used for the determination. A tablet is dissolved in 2 ml of ethanol by gentle heating in the electrolytic cell which is temporarily fitted with a glass tube reflux condenser. The cooled suspension is mixed with 2 ml of an acetate buffer of pH 5, which brings the pH of the mixture to 5·6. After removal of O by a current of N, the wave from − 1·0 to − 1·5 V is recorded at 20°. A tablet containing 5 mg of prednisone (**I**) gives a 75-mm wave; a deduction of 3 mm is made for the effect due to the tablet additives and the base soln. For the determination of 6 to 12 mg of **I**, the vol. of ethanol and buffer soln. must be doubled. Calibration and temp. correction graphs are given. The results are accurate within ± 1%. Prednisolone, for which $E_{\frac{1}{2}}$ lies 0·05 V on the negative side of the value for **I**, can be determined under the conditions described. P. S. ARUP

1903. **A quantitative papergram assay method for prednisolone in tablet formulations.** P. D. Meister, C. A. Schlagel, J. E. Stafford and J. L. Johnson (The Upjohn Company, Kalamazoo, Mich.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (8), 576–579.—This method almost completely eliminates excipients and degradation products which interfere with other methods of assay, and although too tedious for routine use it may be used to establish the reliability of other methods and to assay aged tablets. *Procedure*—Form a slurry of the powdered tablets in H_2O (10 ml) and 25% Na_2CO_3 soln. (2·5 ml) and extract with $CHCl_3$ (50 + 30 + 30 ml). Evaporate the combined extracts to dryness, transfer to a 1-ml flask with $CHCl_3$ - 1:2-dichloroethane (1:1) and dilute to volume. Submit a 0·1-ml aliquot to descending partition chromatography on Eaton Dikeman 613 paper, with propane-1:2-diol - methanol (1:1) as stationary phase and glacial acetic acid - saturated soln. of propane-1:2-diol in toluene (1:50) as mobile phase. After development for 40 hr., process according to the method of Reineke (*cf. Anal. Abstr.*, 1957, **4**, 1943) and measure the u.v. absorption of the methanolic eluate at 224 mμ, 244 mμ and 260 mμ. The calculation provides correction for the small amounts of non-steroidal absorbing substances present in the eluate.

A. R. ROGERS

1904. **Comparison of methods of assay of prednisolone in compressed tablets.** J. G. Wagner, J. K. Dale, C. A. Schlagel, P. D. Meister and R. E. Booth (The Upjohn Company, Kalamazoo, Mich.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (8), 580–584.—A method based on extraction with ethanol and the tetrazolium reaction was unsatisfactory; it gave high values with decomposed tablets and with tablets containing lactose as excipient. The $CHCl_3$-extraction and tetrazolium-reaction procedure described below gave results comparable with those by the paper-chromatographic method of Meister *et al.* (*cf. Anal. Abstr.*, 1959, **6**, 1903) and is suitable for routine assays. *Procedure*—Mix the powdered tablets (containing $\simeq$ 5 mg of prednisolone) with H_2O (4 ml) and add $CHCl_3$ with shaking until the vol. of the lower phase is 500 ml. Evaporate a 20-ml aliquot of the lower phase to dryness. Dissolve the residue in ethanol (20 ml) and add 0·5% ethanolic 2:3:5-triphenyltetrazolium chloride (2 ml) and a filtered mixture of 10% aq. tetramethylammonium hydroxide - ethanol (1:9) (2 ml). Mix, and after 40 min. measure the extinction at 485 mμ. A. R. ROGERS

1905. **Gravimetric estimation of camphor in pharmaceutical preparations via its semicarbazone. II.** K. K. Kaistha (State Drugs Control Lab., Ambala Cantt, Punjab). *Drug Standards*, 1958, **26** (3), 83–85.—The method described previously (*cf. Anal. Abstr.*, 1957, **4**, 4108) has now been applied with slight modifications to the determination of camphor in liniments, spirits, tinctures and pigments. A. R. ROGERS

1906. **Argentimetric determination of hexobarbitone and phenobarbitone.** G. Vastagh and E. Szabolcs (Chem. Dept., State Inst. Hygiene, Budapest). *Arzneimittel-Forsch.*, 1958, **8** (6), 355–358.—It is claimed that, contrary to previous reports in the literature, hexobarbitone and phenobarbitone (**I**) may be determined accurately by argentimetric titration. Exact control of experimental conditions is required, particularly of the pH. If **I** is titrated in alkaline ethanolic soln., alkaloid bases or their hydrochlorides, phenazone, amidopyrine, caffeine and (in very small amounts) bromides do not interfere. A. G. COOPER

1907. **Analysis of some methanesulphonic acid derivatives. I. Iodimetric determination of Novalgin [sodium methyl-(2:3-dimethyl-5-oxo-1-phenylpyrazolin-4-yl)aminomethanesulphonate] and Melubrin [sodium (2:3-dimethyl-5-oxo-1-phenylpyrazolin-4-yl)aminomethanesulphonate] in the presence of phenazone and amidopyrine.** E. Schulek and L. Maros (Inst. für anorg. and anal. Chem., L. Eötvös Univ., Budapest). *Anal. Chim. Acta*, 1958, **19** (1), 4–9.—Former methods of characterising Novalgin and Melubrin by their methylsulphonic groups in the presence of phenazone and amidopyrine have been unsatisfactory. The present method depends on the reaction of the aldehyde bisulphite with alkaline KCN to give sulphite and glycollic acid nitrile. The sulphite in H_2SO_4 soln. is then determined iodimetrically. Potassium iodide is added to the titration mixture to inhibit the reaction of excess of CN^- with iodine. F. J. M.

1908. **Contribution to the analysis of N-butyl-N′-sulphanilylurea [carbutamide].** H. Bräuniger and H. Duda (Pharm.-chem. Inst., Univ. Rostock). *Pharm. Zentralh.*, 1958, **97** (7), 305–310.—The following methods for the characterisation and determination of N-butyl-N′-sulphanilylurea are described—direct and indirect titration with 0·1 *N* $KBrO_3$; titration with 0·1 *M* $NaNO_2$; titration in dimethylformamide soln. with 0·1 *N* Na methoxide; preparation of the Schiff's bases with 11 aliphatic and aromatic aldehydes; titration of the Schiff's bases in dimethylformamide soln. with 0·1 *N* Na methoxide; pptn. of the copper salt of the Schiff's base with salicylaldehyde; absorptiometry of the Schiff's base with salicylaldehyde. The quant. methods of determination are mostly accurate to within ± 0·5%. A. R. ROGERS

1909. **Separation of 1 : 2 : 3-trimercaptopropane [1:2:3-propanetrithiol] from dimercaprol by partition chromatography.** E. G. Rippie, A. A. Kondritzer and R. I. Ellin (Physiol. Div., Directorate of Medical Research, Army Chemical Center, Md.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (7), 529–530.—Form a slurry of silicic acid (20 g) with a 0·1% soln. of sodium bisulphite in 0·2 *M* phosphate buffer of pH 6 (20 ml) and $CHCl_3$ (100 ml). Pack it into a chromatography tube, and wash it free from $CHCl_3$ with light petroleum (boiling-range 35° to 50°) - di*iso*propyl ether (1:1). Add the sample (100 mg) and develop with the same solvent mixture. Recovery of 1:2:3-propanetrithiol is obtained in the first 20 ml of eluate to the extent of 98%, and recovery of the dimercaprol in the next 40 ml of eluate to the extent of 92 to 96%, as shown by titration with iodine. The losses are attributed to oxidation catalysed by trace quantities of Cu in the column. (*Cf.* Ellin and Kondritzer, *Anal. Abstr.*, 1958, **5**, 3141.) A. R. ROGERS

1910. **The use of microscopic and X-ray diffraction methods for the identification of sedatives and anti-convulsants.** W. G. Penprase and J. A. Biles (Sch. of Pharm., Univ. of S. Calif., Los Angeles, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1958, **47** (7), 523–528.—Crystals of 20 of the newer barbiturates, the hydantoins and other sedatives were obtained by fusion, sublimation, crystallisation from 50% ethanol and crystallisation from aq. NH_3 soln. (*d*, 0·880). Photographs of the different crystalline habits show this method to be a useful means of identification. Copper $K\alpha$ X-ray diffraction powder diagrams were made and the *d* distances are reported. A. R. ROGERS

1911. **Argentimetric estimation of acetazolamide.** P. M. Parikh and S. P. Mukherji (Zandu Pharm. Works Ltd., Bombay). *Indian. J. Pharm.*, 1958, **20** (7), 179–180.—The combination of acetazolamide (**I**) with 2 equiv. of $AgNO_3$ in ammoniacal soln. to give an insol. salt forms the basis of a rapid method of assay. The recovery is > 99%. The method may be applied with slight modification to the determination of **I** in tablets; there is no interference by starch, lactose, sucrose, calcium phosphate, gum acacia, talc or magnesium stearate. *Procedure*—Dissolve the sample (200 mg of **I**) in H_2O (50 ml) and dil. aq. NH_3 (5 ml), add 0·1 *N* $AgNO_3$ (25 ml), heat, cool, and filter through sintered glass (porosity 4). Wash the ppt. with H_2O (3 × 10 ml). Acidify the filtrate with dil. HNO_3 and titrate with 0·1 *N* NH_4SCN, with $(NH_4)_2SO_4.Fe_2(SO_4)_3$ as indicator. Perform a blank titration. A. R. ROGERS

1912. **Analysis of 1-ethynyl*cyclo*hexyl carbamate (Valamin) and 1-ethynyl*cyclo*hexyl allophanate (Dolcental).** R. Preusz and R. Kopp (Pharmaceut. Inst., Univ. Freiburg). *Arzneimittel-Forsch.*, 1958, **8** (7), 412–416.—A paper-chromatographic procedure for the identification of the hypnotic agents 1-ethynyl*cyclo*hexyl carbamate and 1-ethynyl*cyclo*hexyl allophanate with ligroin - dioxan as solvent, and optional impregnation of the paper with propane-1:2-diol is described. The test may be performed with from 2 to 10 μg of the compounds, and other hypnotics do not interfere. A. G. COOPER

See also Abstracts—**1606**, Determination of salicylic acid in acetylsalicylic acid. **1610**, Determination of chloramine T. **1651**, Determination of $Hg(OCN)_2$. **1864**, Chromatography and electrophoresis of glycosides. **1876**, Detection of thyroxine. **1940**, **1941**, Determination of folic acid. **1942**, Determination of vitamin B_{12}. **1943**, Determination of ascorbic acid.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

1913. **The time of inversion of sucrose solutions on a boiling-water bath and its effect on sugar determination.** W. Schoch and M. Roulet (Gutsverwaltung u. Landwirtschaftliche Versuchsanstalt,

Lieberfeld-Bern, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1958, **49** (6), 423–433.—Experimental results show that, in the determination of sucrose, the inversion stage if carried out in an HCl-containing soln. at pH 1 to 2 on a boiling-water bath is complete in 10 min. and that the usual inversion time of 30 min. is unnecessarily long. Three methods were studied and reproducible and accurate results (99·604 ± 0·390% on actual content) were obtained with the shorter inversion time. More accurate results are also obtained with the shorter inversion time when sucrose is determined in the presence of maltose and lactose, since the latter disaccharides are unaffected by the treatment.

S. M. MARSH

1914. **Determination of calcium and magnesium in cane juice.** M. C. Bennett and N. O. Schmidt (Dept. of Sugar Chem. & Technol., Imp. Coll. of Tropical Agric., Trinidad). *Int. Sugar J.*, 1958, **60**, 225–230.—It is shown that results for Ca and Mg on a juice clarified with $FeCl_3$ and sodium diethyldithiocarbamate (**I**) agree excellently with those from the ash of the same juice, so that ashing is unnecessary. Treat 100 ml of juice with 5 ml of 4 *N* HCl and 10 ml of 0·5 *M* $FeCl_3$, and after 2 to 3 min. bring to pH 4 to 5 with 6 *N* NaOH, add 1 ml of *M* acetate buffer (pH 4·5), make up to 200 ml and filter. To 100 ml of the filtrate add 100 mg of solid **I**, shake, and filter off the black ppt. To the filtrate add 100 mg of **I** and shake with 10 ml of $CHCl_3$. The extraction of Mn is shown by a purple colour in the $CHCl_3$ layer. Continue the addition of **I** and extraction till the $CHCl_3$ layer remains colourless or faint yellow. Remove Ca from a portion (50 ml) of the aq. layer by adding 10 ml of 0·2 *M* sodium oxalate at pH 5·0, making up to 100 ml, allowing to stand for 2 hr. and centrifuging off the calcium oxalate for 20 min. at 1000 ***g***. Calcium and Mg together in the untreated aq. layer, and Mg alone in the oxalate-treated sample, are determined by titration with EDTA disodium salt) by the usual methods.

R. E. ESSERY

1915. **Colorimetric determination of 5-(hydroxymethyl)-2-furfural in glucose syrup with benzidine.** Yoshiro Yoshihiro and Matao Nakamura (Inst. of Ind. Sci., Tokyo Univ., Yayoi-cho, Chiba). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (8), 972–975.—Benzidine (**I**) is used for the determination of 5-(hydroxymethyl)-2-furfural (**II**) in glucose syrup, but **II** is less sensitive to **I** than furfuraldehyde (McCance, *Biochem. J.*, 1926, **20**, 1110). The extinction at 460 mμ of a butanolic soln. of **II** in the presence of **I** in glacial acetic acid increases with increase in amount of **I**, but the stability of colour decreases. **II** is extracted with *n*-butanol from aq. soln. whilst most of the glucose remains in the aq. phase. The sample (5 ml) is shaken with butanol (5 ml) and an aliquot of the latter (1 to 3 ml) is diluted to 4 ml, and mixed with water (1 ml) and **I** in glacial acetic acid (1%, 5 ml). The mixture is kept at 30° for 15 min. and the extinction measured at 460 mμ. The amount of **II** is calculated by the use of an empirical factor. K. SAITO

1916. **Determination of malic acid in maple syrup by an ion-exchange procedure. An adaptation of the Goodban and Stark method.** C. O. Willits, W. P. Scholette and J. C. Underwood (Eastern Reg. Res. Lab., Philadelphia, Pa., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 658–662.—An ion-exchange procedure based on the method of Bryant and Overell (*Nature*, 1951, **167**, 361) has been applied to Goodban and Stark's method (*Anal. Abstr.*, 1957, **4**, 3161) for the determination of malic acid in plant juices. Other acids normally present in maple syrup do not interfere, and sugars are quantitatively eliminated. The colour developed by malic acid and 2:7-dihydroxynaphthalene in strongly acidified solution accords with Beer's law. An accuracy of ± 0·01% is reported.

A. A. ELDRIDGE

1917. **Analysis and evaluation of malted sweets.** H. Hadorn (Lab. VSK, Basel). *Mitt. Lebensmitt. Hyg., Bern*, 1958, **49** (5), 290–298.—Various manufactures of malted sweets were examined chromatographically and related to the chromatographic findings of malt extract itself and starch hydrolysate. The first showed dextrose, maltose, an oligosaccharide and a trace of sucrose. Sucrose was absent in starch hydrolysate, which, however, contained two additional different oligosaccharides. These were also found in malted sweets. The actual malt-extract content of these sweets could therefore not be assessed by determination of their maltose content. As only malt extract contains P, this could be determined as P_2O_5 and the maltose content derived from malt extract calculated from it. Six varieties of malted sweets were examined and found to contain: sucrose (66·8 to 83·7%), maltose (2·6 to 8%), glucose (5·7 to 10%) and oligosaccharides (8·0 to 14·9%). The P_2O_5 content ranged from 22·8 to 55·0 mg per 100 g. This, if converted to the malt-extract content of the sweets gave 4·1 to 9·2%; a similar calculation based on the maltose gave 5·6 to 15·9%. S. BAAR

1918. **A simple detection of protein in starch.** Jun Mizuguchi, Shuichi Suzuki and Fujio Takahashi (Tokyo Inst. Tech., Meguro-ku, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (8), 1051–1052.—The colour reaction of the potassium salt of tetrabromophenolphthalein ethyl ester (**I**) with protein is applied to the detection of proteins in starch. The indicator is blue (max. absorption 590 mμ) in a basic soln. (pH > 5·5) and yellow (max. absorption 440 mμ) in an acid soln., but in the presence of proteins **I** remains blue in an acetic acid (*e.g.*, 0·1 *N*, 1 ml) soln. The intensity of the blue coloration varies as the amount of N, determined by the Kjeldahl method. K. SAITO

1919. **The standardisation of moisture determination in cereals.** M. Chopin and G. Chopin (Acad. Agric. France, Paris). *Compt. Rend. Acad. Agric. France*, 1958, **44** (3), 154–156.—The varying conditions of humidity of the laboratory atmosphere were found to cause deviations of up to 0·6% in the moisture figures determined by desiccation in the regulation drying cabinet at 102°. To obviate this, an apparatus was designed in which a current of air of known humidity is used. Air from the laboratory is cooled to 0° by means of a thermosyphon, and then re-heated to the working temp. of the cabinet, *i.e.*, 102°. A. TESSLER

1920. **Qualitative determination of volatile fatty acids in dough and bakery products.** A. W. Croes (Inst. voor Graan, Meel en Brood, T.N.O., Wageningen, Netherlands). *Chem. Weekbl.*, 1958, **54** (30), 396–397.—The finely divided sample (200 g) treated with water and conc. H_2SO_4 (5 ml) is diluted to 1 litre, shaken for 45 min. and centrifuged. Of the clear liquid, 600 ml is taken, distilled with 5 ml of

conc. H_2SO_4 on a paraffin bath at 160° into a 500-ml ice-cooled flask, and the distillate is made alkaline with $Ca(OH)_2$ and then evaporated to 5 ml. The cooled product is made acid to litmus paper with dil. H_2SO_4, then filtered, and paper chromatography (Whatman No. 1) is then applied with NH_3-saturated *n*-butanol for 16 hr. The air-dried paper is developed by spraying with chlorophenol red. Formic acid is detected separately with 0·1 *N* $AgNO_3$. P. RENTENAAR

1921. **The analysis of pasta made with eggs.** M. R. Alliot (Lab. Centr. de la Repression des Fraudes, Paris). *Ann. Falsif.*, 1958, **51**, 151–157.—From the existing methods of determination of the amount of egg in pasta, three (*cf.* Hadorn and Jungkunz, *Mitt. Lebensmitt. Hyg., Bern*, 1952, **43**, 1) were found the most suitable and are described in detail. (*i*) The egg content may be calculated from the total lipids, determined by Soxhlet extraction, allowance being made for the fat content of the wheat used in the manufacture of the pasta. (*ii*) The egg content may also be calculated from the cholesterol, determined as digitonide, allowing for the phytosterol of the wheat, or (*iii*) from the amount of water-soluble proteins. *Procedure*—The proteins of the wheat are pptd. by means of $MgSO_4$ at 30° to 32° from an aq. suspension of the sample. The egg albumin passes into the filtrate, where it is coagulated by boiling, subsequently being treated with Fehling's soln., and the N is determined by Kjeldahl's process. From the albumin found, the egg content may be calculated with greater accuracy than by either of the other two methods, but this method cannot, of course, be used if the pasta is made with dried yolk instead of whole eggs. A. TESSLER

1922. **The proximate analysis of rice carbohydrates.** J. R. Fraser and D. C. Holmes (Gov. Lab., Clement's Inn Passage, London). *J. Sci. Food Agric.*, 1958, **9** (8), 511–515.—The four main classes of carbohydrate in rice have been determined separately. (*i*) Starch was estimated polarimetrically in $CaCl_2$ soln. (*ii*) Pentosans were estimated colorimetrically with orcinol - $FeCl_3$ soln. after distillation with 12% HCl. (*iii*) Sugars were titrated with Fehling's soln. (*iv*) Cellulosic material (fibre) was estimated by removal of fat, protein, starch, sugars and pentosans by enzymolysis and weighing the residue. Thus, starch was first solubilised by autoclaving with $CaCl_2$ soln. and the mixture was then digested with trypsin at pH 8·0 for 24 hr. at 40°. The residue was filtered off and treated with 0·06 *N* HCl (150 ml). Pepsin (0·5 g) was added and the soln. was digested for 48 hr. at 40°. Finally the residue was washed and weighed. The whole procedure requires 4 days for completion. The sum of carbohydrates thus found agrees well with the method giving values for "carbohydrates by difference." E. DUX

1923. **Detection of refining of lard.** R. Grau and A. Mirna (Chem.-phys. Inst. der Bundesforschungsanstalt f. Fleischwirt., Kulmbach). *Fette, Seif., Anstrichmitt.*, 1958, **60** (7), 533–534.—From experiments on samples of fresh and rancid lard it was found that the extinctions in the u.v. range of the unsaponifiable fraction of rancid lard are considerably higher than with sound lard, and that this change is not influenced by treatment of the former with bleaching earths (*cf.* Kaufmann *et al.*, *Anal. Abstr.*, 1957, **4**, 3123). A. TESSLER

1924. **Studies on the analysis of soy sauce. I. Determination of glycerol.** Akira Okuhara and Tamotsu Yokotsuka (Noda Shoyu Co., Noda, Chiba-ken). *J. Agric. Chem. Soc. Japan*, 1958, **32** (2), 138–143.—Various methods were examined. The extraction of glycerol from soy sauce is best effected by distillation of the diluted sample at 20 mm of Hg. The glycerol in the distillate is determined by the periodate method.

II. Potentiometric titration of acid ingredients and formol nitrogen. Toshio Sakai and Tamotsu Yokotsuka. *Ibid.*, 1958, **32** (2), 144–151.—For the alkalimetric titration of organic acids in soy sauce (acetic, lactic, succinic, oxalic, propionic, *iso*butyric, *iso*valeric and *n*-hexanoic acids), pH 8·2 is the best end-point. The initial pH for formol nitrogen titration was examined and 8·5 was found to be most satisfactory. The sample is diluted to 50 times its vol., and a 25-ml portion is titrated with 0·05 *N* NaOH to pH 8·2 (organic acid content). After the pH is adjusted to 8·5, the product is mixed with formaldehyde (pH 8·5) to bring the concn. to 9 to 15% and titrated with 0·05 *N* NaOH. Ammonia is determined separately and the amount of corresponding nitrogen is deducted from the titrated value for formol nitrogen. Sugars do not interfere. K. SAITO

1925. **Detection of food preservatives by paper chromatography.** A. C. Cooke (Dominion Lab., D.S.I.R., Wellington, New Zealand). *N.Z. J. Sci.*, 1958, **1** (3), 412–416.—In the method described for detecting preservatives in foods, 20 g of solid macerated with water, or 50 ml of beverage, is acidified, and, after addition of 10 ml of ethanol, is extracted with $CHCl_3$; the solvent is evaporated, and the residue is dissolved in ethanol and chromatographed on paper, with *n*-butanol - ethanol - 3 *N* aq. NH_3 - 3 *N* ammonium carbonate (40:11:9·5:9·5) as solvent mixture. Fluorescent spots are detected by the u.v. fluorescent screen technique, and outlined with pencil, and the paper is then sprayed with diazotised *p*-nitroaniline, followed by Na_2CO_3 soln.; this colours *m*- and *p*-hydroxybenzoic acids red, vanillic acid purple, vanillic esters mauve, salicylic acid orange, thiourea yellow, and some other preservatives pink. Aliphatic acids do not interfere. The R_F values of 19 preservatives and 8 food constituents are listed. M. D. ANDERSON

1926. **Testing of plastics for use with foodstuffs.** A. C. Case (Whitbread & Co., Ltd., Chiswell St., London, England). *J. Inst. Brewing*, 1958, **64** (4), 289.—The attention of food and beverage manufacturers and packers is drawn to a report of The British Plastics Federation ('Report of the "Toxicity" Subcommittee of the Main Technical Committee,' London, 1958) in which a method is proposed for assessing the toxicity of plastics, plasticisers and stabilisers. A "toxicity quotient" is calculated from known toxicity data combined with the degree of extraction obtained under defined conditions. At present the scheme is applied to rigid and pliable plastics used for lining vessels, piping and packages, but specifically excludes can-linings. It is suggested that the proposed methods could usefully be applied to these, and also to the testing of plastics for possible effects on the palate. R. E. ESSERY

1927. **Identification of stored-products insects by the micromorphology of the exoskeleton. VIII. Moth larvae.** L. G. Ensminger (Food and Drug

Admin., Dept. of Health, Education & Welfare, Los Angeles, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 704–738.—Insect fragments of diagnostic value are described and illustrated.

A. A. ELDRIDGE

1928. **Conductimetric analysis of hops. Report by the Dutch Association of Brewery Chemists.** Abstr. from *Brouw. en Mout*, 1956/7, **16** (6), 207, *Brauwissenschaft*, 1958, **11** (9), 212–213.—This procedure consists in the extraction (in a Turmix mixer) of the sample (15 g) with benzene or hexane (100 ml) and the conductimetric titration of 10 ml of the extract, mixed with 40 ml of methanol, with a standard methanolic soln. of Pb acetate (4 g of Pb acetate trihydrate in 100 ml). The results thus obtained for the α-acids have been shown by collaborative analyses (in seven laboratories) to be in satisfactory agreement with those obtained by the Govaert-Verzele method, and in fair agreement with the results obtained by the method of Alderton *et al.* For the extraction of the α-acids, benzene has proved more suitable than hexane. The Pb acetate soln. must be freshly prepared before use. The results show a standard deviation of $\pm$ 0·16 for 10 determinations.

P. S. ARUP

1929. **A note on the determination of the peroxidase content of hop plants.** E. W. Weston. *Ann. Rep. Dep. Hop Res., Wye College*, 1957, 92–97.—The Tatchell method (*cf. Ibid.*, 1952, 59) is modified to enable the determination to be carried out by one colorimetric measurement. *Procedure*—Wash 1 g of the dried and ground hop cones with ether to remove the resins, and transfer the residue to a 100-ml flask with a Na acetate - acetic acid buffer (pH 4·3). Make up to 100 ml, shake for 15 min., and filter. To 50 ml of the filtrate add 5 ml of 1% *o*-phenylenediamine soln. and 5 ml of 2% H_2O_2, and then set aside for 90 min. at room temp. For the measurement of the optical density (with an Ilford blue 602 filter) take 5 ml of the soln. diluted with acetone to 50 ml. The coloration thus produced is proportional to the content of peroxidase. No significant correlation is found between the peroxidase content and the ratio of α- to β-resins.

P. S. ARUP

1930. **Proposed non-ashing technique employing ion-exchange resins for the determination of lead in wine.** R. A. Edge and N. Penny (Gov. Chem. Lab., Cape Town, S. Africa). *J. Sci. Food Agric.*, 1958, **9** (7), 401–403.—In the proposed method, 50-ml aliquots of the wine, freed from solid particles, are run through a column containing 30 g of Dowex-50 resin that has been washed with boiling 4 *N* HCl. The Pb, together with other cations, is eluted with 4 *N* HCl, and is determined colorimetrically with dithizone. Other cations normally present in wine do not interfere. The results, although not so consistent, compare favourably with those given by the iodine pentoxide method of Greenblau and van der Westhuyzen (*Anal. Abstr.*, 1957, **4**, 273) at concn. of $\simeq$ 2 p.p.m. of Pb, but tend to be higher at lower concn. The method, which requires $\simeq$ 3 hr., might find use in control work, to establish whether the concn. of Pb is less than the statutory limit of 1 p.p.m.

J. H. WATON

1931. **New micro-methods for the determination of some of the principal ions in fermented liquids.** F. Mecca and F. Calabrese (Milan Univ., Italy). *Chim. e Ind.*, 1958, **40** (7), 537–543.—A range of colorimetric and nephelometric methods for the determination of various ions in wines, other fermented liquors and vinegars has been developed. Total Fe, Fe^{2+}, Fe^{3+}, Cu^{2+}, Zn^{2+}, PO_4^{3-} and Ca^{2+} are determined on the original liquid. A sample is then ashed, the residue taken up in dilute acetic acid and Na^+, K^+, Cl^-, Mg^{2+} and SO_4^{2-} are determined. Ashing in the presence of HNO_3 enables the combined SO_4^{2-} and SO_3^{2-} contents to be obtained. From the concn. of the various ions, indications can be obtained of some of the treatments, *e.g.*, de-acidification, sulphiting, phosphating or ion exchange, which the products may have undergone.

L. A. O'NEILL

1932. **Chromatographic and conductimetric determination of volatile fatty acids.** R. Jolivet (Fac. Pharm., Algiers). *Ann. Falsif.*, 1958, **51**, 158–159.—The method of Hiscox and Berridge was modified in three respects. (*i*) Sections of the chromatogram, containing the separated ethylamine salts of the fatty acids, were cut out, separate aq. soln. were prepared and the acids determined by conductimetry. (*ii*) To prevent serious losses due to dissociation and evaporation, all drying operations were carried out in an atmosphere containing ethylamine vapours. (*iii*) To preserve the colours, the chromatogram was treated with a soln. of plastic. With the method thus modified it was possible to detect in various fermentation products (such as silage, wines, etc.) certain fatty acids whose presence there had not been suspected.

A. TESSLER

1933. **Radiometric estimation of unsaturated fatty acids on chromatograms.** A. Z. Budzynski, Z. J. Zubrzycki and I. G. Campbell (Dept. of Radiochem., Inst. Nucl. Res., Warsaw, Poland). *Nature*, 1958, **182**, 178–179.—The chromatogram is prepared on S. & S. 598*L* paper (280 mm × 15 mm) by ascending reversed-phase chromatography under N, with a mixture (*A*) of synthetic hydrocarbons (boiling-range 180° to 190°, bromine number 0·16) as stationary phase and 90% acetic acid saturated with *A* as mobile phase. The strip is dried at 60° for 1 hr. in N and is then immersed for $\simeq$ 150 min. in 6·5 ml of a soln. of 0·0022 *M* NaI, 0·0044 *M* $NaIO_3$, with $\simeq$ 5 μC of ^{131}I per ml (as iodide), and subsequently made 0·025 *M* in H_2SO_4. The strip is removed and dried for 12 hr. at 20° to remove the excess of unbound ^{131}I. The distribution of β-activity is then determined by passing the strip, in 2-mm stages, under a slit (15 mm × 2 mm) in a Perspex-glass screen (4 mm thick) placed under an end-window Geiger - Müller counter. The curves of spot-content (μg of acid) *vs.* either integrated spot-activity or max. spot-activity (≡ spot length) are linear for chromatograms of oleic and linoleic acids. The method can be calibrated by concurrent chromatography of known amounts of the pure acids.

W. J. BAKER

1934. **Paper chromatography in the study of fats. XXVI. The quantitative paper chromato - polarographic analysis of fatty acids.** H. P. Kaufmann and M. M. Deshpande (Inst. f. Pharm. u. LebensmittChemie, Univ. Münster). *Fette, Seif., Anstrichmitt.*, 1958, **60** (7), 537–541.—The fatty acids are separated from one another by paper chromatography and their copper salts obtained by immersing the whole chromatogram in Cu acetate soln. The strip is then cut into separate sections (corresponding to the individual copper soaps), each of which is treated with dil. H_2SO_4. The resulting solutions of $CuSO_4$ are determined quant. by polarography and

the amounts of the original fatty acids calculated. The method was tried out on known amounts varying from 100 to 400 μg of pure fatty acids and their mixtures, and also on natural fats. The maximum error of the determination is ± 5%, which compares favourably with that of other micro-methods. A. TESSLER

1935. **Separation of saturated and unsaturated fatty-acid esters by gas - liquid chromatography.** B. M. Craig and N. L. Murty (Prairie Regional Lab., Saskatoon, Saskatchewan, Canada). *Canad. J. Chem.*, 1958, **36** (9), 1297–1301.—Methyl esters of fatty acids can be separated according to chain-length on a column (12 ft. × 0·25 in.) of washed silicone-grease supported on C_{22}-firebrick (20 to 40 mesh) and, according to both chain-length and degree of unsaturation, on a column (6 ft. × 0·25 in.) of adipic or succinic plasticiser (or the two in series) on the same support. The materials are packed in copper columns, which are operated at 210° with He as carrier; samples are from 3 to 15 μl, and flow rates from 70 to 100 ml per min. By combining the two procedures, the component fatty acids in fats and oils can be determined. Results for methyl esters of fatty acids from tallow, lard, soya-bean and herring oils are reported. Approx. parallel linear curves for log. emergence time *vs.* C number are obtained for saturated, mono-, di- and tri-ethenoid methyl esters. The succinic plasticiser gives a more effective separation of stearic and oleic esters, and the adipic column of linolenic and arachidic esters. W. J. BAKER

1936. **Paper chromatography of higher fatty acids.** A. Kopecký (Res. Inst. of Fats and Oils, Prague). *Průmysl Potravin*, 1958, **9** (7), 385–386.—Benzene soln. of fatty acids (0·005 to 0·01 ml) were placed on Whatman No. 2 paper impregnated with a 15% soln. of liquid paraffin in light petroleum and separated with the use of 90% acetic acid saturated with liquid paraffin as solvent. The dry chromatograms were immersed in a soln. of Pb acetate (1%), washed in H_2O and moistened with a soln. of Na_2S (1%); dark-brown spots appeared. The unsaturated acids can be detected by the use of iodine vapour. The procedure described has been used successfully for the determination of fatty acids in fats, oils, and fatty stocks used for the production of edible fats and of soaps. J. ZÝKA

1937. **Emission spectrographic detection of trace metals in fats.** K. Täufel and K. Barthel (Inst. f. Ernähr. in Potsdam-Rehbrücke, Dtsch. Akad. d. Wissensch., Berlin). *Fette, Seif., Anstrichmitt.*, 1958, **60** (7), 534–536.—A progress report is presented, describing work on the detection and determination, in fats, of trace metals, especially Fe, Ni and Cu. To ensure even combustion, the sample is mixed with dioxan or methyl oleate and is continuously atomised, by means of compressed air or oxygen, along a channel bored through the lower electrode, into the spark. In experiments on synthetic soln., as little as 0·5 μg of Cu, 5 μg of Fe and 5 μg of Ni per ml of methyl oleate could be detected by this rapid method. A. TESSLER

1938. **Determination of the peroxide value of edible fats by colorimetric iodimetric procedures.** P. A. T. Swoboda and C. H. Lea (Low Temp. Sta. for Res. in Biochem. and Biophys., Univ., Cambridge). *Chem. & Ind.*, 1958, (33), 1090–1091.—Iodine liberated by the peroxide is converted into a tri-iodide or starch complex, the extinction of which is measured. *Procedure*—The sample is dissolved in 3 ml of acetic acid - $CHCl_3$ (3:2) and de-aerated with CO_2. Iodide reagent (1·2 g of KI in 1 ml of H_2O) (0·08 ml) is added and the tube is set aside for 1 hr. Cadmium acetate soln. (0·5%) (7 ml) is added, the aq. layer is separated by centrifuging, and its extinction is measured at 350 mμ. Alternatively, 6 ml of the Cd acetate soln. and 1 ml of 10% aq. starch - urea soln. are added and the extinction is measured at 550 mμ. Calibration is effected using 0·0002 *N* KIO_3 in 0·5% Cd acetate soln. Results obtained are within 2% of the theoretical and comparable with volumetric results. E. J. H. BIRCH

1939. **Quantitative determination of traces of free gossypol in fats, oils and fatty acids by paper chromatography.** G. Schramm and J. H. Benedict (Procter & Gamble, Cincinnati, Ohio, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1958, **35** (7), 371-373.—About 10 g of the sample dissolved in heptane is extracted with 2 × 25 ml of dimethylformamide - water (2:1) and the combined lower layers are further extracted with heptane - $CHCl_3$ (9:1) (3 × 50 ml) and centrifuged. The combined upper layers are then dried over anhyd. Na_2SO_4, filtered and concentrated to 5 ml. This soln., together with gossypol standards, is applied to a chromatographic strip and developed by the ascending method with the solvent heptane - $CHCl_3$ - acetic acid (80:20:5). After 90 min. operation, the strip is air-dried and sprayed with phloroglucinol soln. (2 g in 10 ml of 95% ethanol and 5 ml of conc. HCl) when the colour develops in 1 to 5 min. at room temp. The method is specific for free gossypol, with a lower limit of detection of 10 p.p.m. G. R. WHALLEY

1940. **Assay of folic acid in presence of iron salts.** R. C. Shah, S. B. Gandhi and V. S. Kanetkar (Mac Laboratories Private Ltd., Bombay). *Indian J. Pharm.*, 1958, **20** (7), 180–182.—To overcome interference with the determination of folic acid by the absorptiometric method of the U.S.P. XIV caused by ferrous gluconate (< 1200-fold excess) or ferric ammonium citrate (< 200-fold excess), mask the Fe by complex formation with EDTA. For interference by exsiccated ferrous sulphate (<2000-fold excess), remove the Fe by pptn. with Na_3PO_4 soln. A. R. ROGERS

1941. **Colorimetric determination of folic acid in presence of large amounts of vitamin C.** R. C. Shah, P. V. Raman and S. B. Gandhi (Mac Laboratories Private Ltd., Bombay). *Indian J. Pharm.*, 1958, **20** (7), 184.—To overcome interference with the determination of folic acid by the method of the U.S.P. XIV caused by ascorbic acid (< 1000-fold excess), increase the concn. of $NaNO_2$ soln. to 1% and the concn. of ammonium sulphamate soln. to 5%. A. R. ROGERS

1942. **Estimation of vitamin B_{12} by the colorimetric determination of cobalt.** M. A. H. Sharif (C.S.I.R., Karachi, Pakistan). *Pakistan J. Sci. Ind. Res.*, 1958, **1** (2), 160–164.—The cobalt content of a soln. of vitamin B_{12} was determined by means of nitroso-R salt reagent according to the method used by Kidson and Eskew (*N.Z. J. Sci. Tech., B*, 1940, **21**, 178). The cobalt complex obtained was measured colorimetrically at 550 mμ. For a 2% accuracy, 4 μg of Co, which is equivalent to 100 μg of the vitamin, is required. K. R. C.

1943. **Bromate titrations. III. The determination of ascorbic acid with bromate.** L. Szekeres, E. Sugár and É. Pap (Dept. Agric. Chem., Univ., Budapest). *Z. anal. Chem.*, 1958, **163** (1), 32–34.—Starch - potassium iodide can be used as an indicator in the titration of ascorbic acid with standard bromate soln. A sluggish end-point is obtained if the test soln. contains < 3% (w/w) of HCl. *Procedure*—To the test soln. (10 ml) add conc. HCl (10 ml), water (80 ml), 0·5% starch soln. (1 ml) and 0·1 *N* KI (1 drop). Titrate with 0·1 *N* $KBrO_3$ to a blue end-point. W. T. CARTER

1944. **The tocopherol contents of some Australian cereals and flour milling products.** E. L. Mason and W. L. Jones (W. S. Kimpton & Sons, Melbourne, Australia). *J. Sci. Food Agric.*, 1958, **9** (8), 524–527.—The sample is extracted with diethyl ether. The non-saponifiable fraction of the extract is purified and chromatographed (*cf.* Green *et al.*, *Ibid*, 1955, **6**, 274). A preliminary development is carried out with *cyclo*hexane for 90 min. The paper is then impregnated with 3% of liquid paraffin in light petroleum. After replacement of the solvent by 81% ethanol - water, the chromatogram is developed further for 3 hr. The paper is then sprayed with 2:2′-dipyridyl - $FeCl_3$ soln. The spots, when visible, are cut out and placed in methanol for 15 min., 2:2′-dipyridyl soln. (0·5%) and $FeCl_3$ soln. (0·2%) are then added consecutively and the optical density is recorded at 515 mμ. E. DUX

See also Abstracts—**1605**, **1608**, Determination of ascorbic acid. **1782**, Determination of fructose. **1784**, Determination of amylose in starch. **1836**, Determination of heavy metals in food dyes. **1849**, Determination of ^{137}Cs in foods. **1850**, Determination of ^{89}Sr and ^{90}Sr in fish.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

1945. **Catalyst for determining total hydrocarbons in air.** V. P. Vendt and T. A. Lebedeva (Kiev Inst. of Work Hygiene and Occupational Diseases). *Zavod. Lab.*, 1958, **24** (7), 818–819.—The catalyst is prepared by soaking pieces of pumice in saturated $(NH_4)_2Cr_2O_7$ soln. and then igniting in a muffle-furnace, first at 400° and then for at least 3 to 4 hr. at 800°. It is used in a combustion tube at 700° to 800°. The catalyst is more active than a platinum spiral or CuO for oxidation of all types of hydrocarbons. If CO is present it must be determined separately and allowed for. G. S. SMITH

1946. **A rapid determination of a low concentration of ammonia and carbon dioxide in air by means of detector tubes.** Yoshitaka Kobayashi, Akio Matsumoto and Takeshi Yamada (Fac. of Engng, Yokohama Univ., Minami-ku). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (5), 525–527.—*Procedure* (*for* NH_3)—Air (100 ml) containing 0·002 to 0·7% of NH_3 is passed at 100 ml per sec. through a detector tube (diam. 3 mm) containing colourless methacrylate resin (40 to 80 mesh, 0·15 g) impregnated with ethanolic thymol blue soln. (0·05%, containing 2 drops of conc. H_2SO_4 per 10 ml) (10 ml for 100 g of the resin). A yellow layer develops, the length of which is proportional to the concn. of NH_3. There is no interference from CO_2, O, N, CH_4, SO_2 or acetic esters. Amines produce a similar coloration; Cl (> 0·03%) changes the colour to grey. *Procedure* (*for* CO_2)—When the sample of air (100 ml) containing 0·02 to 0·3% of CO_2 is passed at 100 ml per 200 sec. through a tube (diam. 2 mm) containing activated alumina (150 to 200 mesh, 0·1 g) impregnated with 0·4 *N* NaOH coloured with thymolphthalein (60 ml for 100 g of Al_2O_3), a pink layer is formed, the length varying with the concn. of CO_2, which may be determined by the use of an empirical curve. Strong acid vapour such as HCl, SO_2, Cl_2 or ClO_2 is adsorbed on a very thin layer of alumina near the gas inlet, and does not vitiate the estimation. Hydrogen cyanide is removed by passage through a silica gel layer containing methyl orange and $HgCl_2$; H_2S is absorbed by a similar layer containing $Pb(NO_3)_2$. K. SAITO

1947. **Determination of organic phosphorus insecticides in air.** M. A. Trotsenko and V. P. Vendt (Kiev Inst. of Work Hygiene and Occupational Diseases). *Zavod. Lab.*, 1958, **24** (7), 820–821.—The air is passed over silica gel for adsorption of the insecticide, the gel is extracted with ether, the soln. is evaporated, the residue is decomposed with HNO_3 and $KMnO_4$, etc., and the P is determined colorimetrically by the molybdenum blue method. G. S. SMITH

1948. **Extraction and analysis of gases dissolved in water.** M. Bastick and H. Guérin (Lab. de Chim., Min. Industr., Fac des Sci., Nancy, France). *Bull. Soc. Chim. France*, 1958, (8–9), 1119–1122.—A sample of mineral water (≃ 250 ml) is taken in a bulb closed at either end with a tap and provided with a ground joint to connect it to a round bottomed flask connected to two traps which can be immersed in solid CO_2 - ethanol baths. The apparatus below the bulb is evacuated and the water admitted to the flask for about 2 hr. Evolved gases are collected over mercury. Complete extraction of the gas is ensured by distilling the water into the traps. The gas is analysed largely volumetrically by classical methods. Hydrogen sulphide is determined either by absorption on $CuSO_4$ or preferably iodimetrically in the gas absorbed by KOH soln. The errors inherent in the Winkler method for oxygen in natural waters are avoided by this method. E. J. H. BIRCH

1949. **Note on the determination of low concentrations of sulphate in rain and other waters.** J. K. Johannesson (Wellington City Council Lab., New Zealand). *N. Z. J. Sci.*, 1958, **1** (3), 423–424.—The water is passed through a column of Amberlite IR-120 (H form) to remove cations; it is then boiled to remove CO_2 derived from bicarbonate, and, after cooling, HCl and excess of solid HgO are added. Conductivity, determined at 30°, is almost entirely due to sulphate, a small correction being made for chloride. It may be necessary to correct also for nitrate. The method is rapid, and more accurate than previous methods for concn. of 1 to 10 p.p.m. of sulphate. M. D. ANDERSON

1950. **Automatic analysis [of water] for silica and hardness.** G. Schneider (Suncoast Instrument Div., Milton Roy Co., Philadelphia, Pa.). *Wat. & Sewage Wks*, 1958, **105** (7), 270–272.—A colorimetric analyser designed particularly for controlling boiler water is described. The hardness analyser makes a complete analysis every 6 min. and the silica analyser every 12 min. The difference in light transmittance between the zero cell and the sample cell

is measured by photo-electric tubes in a modified Wheatstone bridge circuit. The silica analysis is based on the molybdosilicate method of Robison *et al.* (*Amer. Soc. Mech. Engrs, Paper No.* SI–A–92). The colorimetric analysers are of standard ranges, 0 to 3 mg and 0 to 5 mg per litre, calculated as $CaCO_3$, with sensitivities of 0·3 and 0·05 mg per litre, respectively. The silica analysers are of standard ranges 0 to 0·5 and 0 to 0·05 mg per litre calculated as SiO_2 and are accurate to within ± 1% and ± 5%, respectively. O. M. WHITTON

1951. **Investigation of a method to determine selenium in water.** R. Červenka and M. Korbová. *Coll. Sci. Rep.*, 1952–1955, *Inst. Hyg. Prague*, 1957, (6).—In order to develop a reliable method for the determination of small quantities of Se in water, certain modifications have been made to the method of Salas [*Summ. Curr. Lit. Wat. Pollut.*, 1948, (21), Abstr. No. 1077]. The final potentiometric titration with thiosulphate was found to be unsatisfactory when the water sample contained only a small quantity of Se, since the potentiometric curves were flattened. A further error was introduced by the gradual formation of a film of Se on the electrodes. The potentiometric titration has therefore been replaced by a polarographic determination in an acid medium.

WAT. POLLUT. ABSTR.

See also Abstracts—**1617**, Spectrographic analysis of water. **1701**, Determination of CO_2 and SO_2 in air. **1727**, Determination of HF in air.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

1952. **Critical evaluation of some colorimetric methods for the determination of phosphorus from the point of view of agrochemical investigations.** J. Sarkadi (Res. Inst. for Soil Sci. and Agric. Chem., Hungarian Acad. of Sci., Budapest). *Magyar Kém. Foly.*, 1958, **64** (7–8), 243–245.—Methods were particularly studied in which ammonium molybdate and *p*-methylaminophenol (metol) or $SnCl_2$, or $SnCl_2$ and metol, or ammonium vanadate are used. They have been compared from various points of view (stability of the complex, effect of interfering ions, etc.) and the findings are tabulated. It was found that, besides the usual factors which influence the determinations (*e.g.*, temperature, pH), others have also to be considered; thus if $SnCl_2$ is used, the concn. of P and of other ions affects the light absorption of the complex. Thus when carrying out a series of analyses on soil- or manure-extracts, the salt content of the unknown and the standard soln. should be chosen so that the difference of the concn. in the samples becomes insignificant. By suitable masking, the effect of interfering ions (*e.g.*, Si, F, Fe) can be eliminated or decreased.

A. G. PETO

1953. **The use of 8-hydroxyquinoline and selenous acid in determining available phosphorus.** M. O. Ghani and A. Islam (Dacca Univ., Pakistan). *Soil Sci.*, 1957, **84**, 445–451.—The investigations were carried out with special reference to lateritic soils of high fixing capacity. Truog's method (*J. Amer. Soc. Agron.*, 1930, **22**, 874), the acetic acid method, and a combination of the two methods were investigated and compared. The fixing capacity of the four oxides decreases in the order Fe_2O_3, Al_2O_3, MnO_2, TiO_2. The use of a combination of 8-hydroxyquinoline and selenous acid is more effective than their individual use for complete fixation of phosphorus during acid extraction. The order of efficiency of methods in extracting chemically available phosphorus is 8-hydroxyquinoline and selenous acid together; selenous acid; 8-hydroxyquinoline; Truog's method; acetic acid method.

K. R. C.

1954. **The derivative thermogravimetric analysis of peat and peat constituents.** F. Paulik and M. Weltner (Inst. for Gen. Chem., Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1958, **16** (2), 159–184 (in German).—A wide range of plant constituents (including starch, cellulose and lignin) has been examined by derivative thermogravimetric techniques. It has been found possible to identify, by this means, the successive products evolved on destructive distillation of peat. Examples are given for peats, humic acid and plant residues from four sources. T. R. ANDREW

1955. **Radiometric determination of potassium in mineral fertilisers.** S. I. Krichmar and L. G. Kalstra (Dnieprodzerzhinsk Nitrate Fertiliser Works). *Zavod. Lab.*, 1958, **24** (8), 925–927.—Apparatus is described for determining K in salts from their natural radioactivity. G. S. SMITH

1956. **Variability in individual core analysis of samples of a 10-0-10 and 8-0-8 fertiliser drawn by several sampling devices.** J. G. A. Fiskell, W. H. Kelly, J. M. Myers, R. C. Crooks, R. Dixon and J. J. Taylor (Univ. Florida, Gainesville, Fla., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 640–649.—Florida sampling tubes (open-end, conical) and Indiana sampling tubes (with solid cone tips) of various dimensions, and a wedge cup for mixer- or belt-discharge samples were employed. When sample preparation is adequate, a 0·7-g sample of a 10-0-10 pulverised fertiliser is satisfactory for determinations of N and K. Sampling at the mixer discharge and belt discharge gave a representative sample, but individual cores showed high variability. The results are considered statistically. Florida tubes were preferred to Indiana tubes for sampling 10-0-10 fertilisers; with 8-0-8 fertiliser the tubes gave the same variability. A. A. ELDRIDGE

1957. **Occurrence of manganese, copper, zinc, molybdenum and cobalt in phosphate fertilisers and sewage sludge.** L. J. Clark and W. L. Hill (Agric. Res. Service, U.S. Dept. Agric., Beltsville, Md., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 631–637.—The ranges of values, in p.p.m., for phosphate rock of various localities of origin, are Mn 15 to 2120, Cu 0·6 to 394, Zn 4 to 1045, Mo 0·7 to 58, and Co 0·6 to 11·6. Results for superphosphate and ammonium phosphate are also recorded. Sewage sludges contained Mn 60 to 790, Cu 100 to 1980, Zn 610 to 3700, Mo 2·1 to 118, and Co 3·4 to 30·5 p.p.m. A. A. ELDRIDGE

1958. **Determination of urea in mixed fertilisers.** W. A. Morgan and E. F. Harford (Polychem. Dept., E. I. du Pont de Nemours & Co. Inc., Wilmington, Del., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 637–639.—The rapid volumetric urease method (Fox and Geldard, *Ind. Eng. Chem.*, 1923, **15**, 743) is applicable with satisfactory precision and accuracy to phosphatic fertiliser mixtures containing 1 to 10% of urea. A. A. ELDRIDGE

1959. **Determination of diphenylamine residues on apples.** H. E. Harvey (Dominion Lab., D.S.I.R., Wellington, New Zealand). *N. Z. J. Sci.*, 1958, **1** (3), 378–382.—Diphenylamine residues on apple peel are extracted with light petroleum, separated on a column of MgO, and eluted with benzene - light petroleum (1:1). Residues in apple pulp are extracted with light petroleum after blending with 70% acetone; water and NaOH are added to the extract, which is then steam-distilled; the distillate is extracted with light petroleum. Diphenylamine is determined by treatment with H_2SO_4 - V_2O_5 reagent, centrifuging, and reading the extinction of the H_2SO_4 layer at 590 mμ. Beer's law is obeyed over the range 10 to 80 μg. The recovery of added diphenylamine was 87 to 106% from pulp, and 81 to 91% from peel.
M. D. ANDERSON

1960. **A one-shot, two-filter application of the Schechter method for mixed DDT and chlorobenzilate spray residues.** F. J. Roth (Calif. State Dept. Agric., Sacramento, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 662–663.—The colour finally developed in the method of Pontoriero and Ginsburg (*cf. Anal. Abstr.*, 1954, **1**, 1992) for the determination of DDT in spray residues is sometimes purplish instead of blue. The admixed red colour is probably not due to the presence of methoxychlor, but may be due to that of chlorobenzilate. Readings are therefore first made with a No. 66 (red) filter to determine the DDT, and afterwards with a No. 54 (green) filter, which measures chlorobenzilate plus DDT interference. The extent of the DDT interference, determined graphically, is subtracted. Sprayed pears afforded results as follows—DDT 0·5 to 2·7, chlorobenzilate 0·2 to 1·5 p.p.m.
A. A. ELDRIDGE

1961. **Thiometon. Its behaviour in plants and the determination of spray residues.** O. Jucker (Agrochem. Lab. Sandoz, Basel). *Mitt. Lebensmitt. Hyg., Bern*, 1958, **49** (5), 299–322.—Thiometon (OO-dimethyl S-ethylthioethyl phosphorothiolothionate) and its oxidation products showed very little cholinesterase inhibition. The compounds were separated by reverse phase partition paper chromatography. The paper was impregnated with Dow-Silicone No. 550, and ethanol - acetone - water (1:1:2) or water alone or the Bush solvent B_1 (*Biochem. J.*, 1952, **50**, 370) was used as the mobile phase. The chromatograms were developed with Br vapour and fluorescein. Only the oxidation products of thiometon were found in the plant. Spray residues on a pear crop at the time of the harvest were < 0·05 p.p.m. of thiometon sulphoxide. The chromatographic method of analysis is accurate to 4 μg ≡ 0·04 p.p.m.
S. BAAR

See also Abstracts—**1688**, Determination of PO_4^{3-} in superphosphates. **1932**, Determination of volatile fatty acids in silage. **1947**, Determination of org. phosphorus insecticides in air. **1962**, The fertiliser flask; use and abuse.

5.—GENERAL TECHNIQUE AND APPARATUS

General

1962. **The fertiliser flask: use and abuse.** J. A. Brabson and W. D. Wilhide (Div. Chem. Development, T.V.A., Wilson Dam, Ala., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1958, **41** (3), 629–630.—Volumetric flasks used for digestion of fertiliser samples with acid are attacked by the HF liberated. The resulting volume error is linear with the number of digestions. After 150 digestions the error caused in the determination of P_2O_5 in KH_2PO_4 was 0·27 to 0·33%.
A. A. ELDRIDGE

1963. **A burette for reductimetric titrations.** M. Matrka, B. Smetana and Z. Ságner (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1958, **8** (7), 367.—A simple automatic burette in which the volumetric soln. is protected by an atmosphere of hydrogen is described. A soln. of $TiCl_3$ when kept in this burette does not change its factor over 4 weeks.
J. ZÝKA

1964. **Filtration attachment for pipettes.** Huo-Ping Pan (Dept. of Food Tech., M.I.T., Cambridge, Mass., U.S.A.). *Mikrochim. Acta*, 1958, (5), 713–714 (in English).—A filtration device permits the removal of a clear liquid from a suspension and also allows liquid to be 'squeezed' from the absorbent by pressing the absorbent.
D. F. PHILLIPS

1965. **Apparatus for the viscometry of organic liquids at high temperature.** W. G. Burns, B. Morris and R. W. Wilkinson (A.E.R.E., Harwell, Berks., England). *J. Sci. Instrum.*, 1958, **35** (8), 291–293.—The viscometer is a suspended level glass-capillary type which is evacuated and sealed to exclude air. During use the viscometer is immersed in a molten salt mixture.
G. SKIRROW

See also Abstract—**1670**, Apparatus for determination of CO.

Chromatography, ion exchange, electrophoresis

1966. **Chromatographic data.** *J. Chromatography*, 1959, **2** (1), Suppl. D1–D8.—Tables of chromatographic data are given for—cholesterol and cholesteryl esters (J. A. Labarrère, J. R. Chipault and W. O. Lundberg); phospholipids (R. F. Witter, G. V. Marinetti, L. Heicklin and M. A. Cottone); organic mercury compounds (J. Kanazawa, K. Koyama, M. Aya and R. Sato); amino acid - copper complexes (M. Beck, J. Csaszar and T. Huska); ε-aminocaproic acid and caprolactam (K. Czerepko); coal-tar food colours (J. Crossley and J. D. R. Thomas); hydrolysis products of organophosphate insecticides (F. W. Plapp and J. E. Casida); 2:4-dinitrophenylhydrazones of some aliphatic carbonyl compounds (E. Sundt and M. Winter); 2:4-dinitrophenylhydrazones of some aromatic compounds (E. Sundt and M. Winter); condensed aromatic compounds and their oxidation products (J. Gasparič); some naphthaquinones, hydroxyanthraquinones and related compounds (J. Gasparič); some quinones (J. Gasparič); alkaloid drugs (in urine) (M. Bühler); relative retention volumes of terpenes and non-cyclic unsaturated alcohols and esters (gas chromatography) (E. Bayer, G. Kupfer and K.-H. Reuther); relative retention volumes of phenols (gas chromatography) (J. Janak and R. Komers); relative retention volumes of phenols (gas chromatography) (G. Bergmann and D. Jentzsch). References to the original publications are given.
N. E.

1967. **Errors of chromatographic analysis connected with incomplete separation.** A. A. Zhukhovitskiĭ and N. M. Turkel'taub (Sci. Res. Geological Prospecting Petroleum Inst.). *Zavod. Lab.*, 1958, **24** (7), 796–798.—A mathematical consideration is

presented of the errors that can occur when the curve obtained does not return to the base line between peaks representing the components of a mixture.
G. S. SMITH

1968. **Compact and versatile fraction collector.** A. Snow, F. A. E. Porter and A. W. Tomalin (Low Temp. Station, Univ. of Cambridge). *J. Sci. Instrum.*, 1958, **35** (7), 243–244.—The tubes are arranged spirally on a horizontal turn-table and each is brought directly under the column to avoid mixing of successive drops in any connecting tube. The fall of a drop can be detected either photoelectrically or mechanically. The detector output operates a counter using cold cathode decay tubes, a relay operating when a pre-determined count is reached.
G. SKIRROW

1969. **Use of fluorescence in chromatography.** N. M. Zyuskin (Kiev Forensic Sci. Res. Inst.). *Zavod. Lab.*, 1958, **24** (7), 793–795.—Methods of studying fluorescence of chromatograms on paper, alumina, etc., in the far and near infra-red regions are described.
G. S. SMITH

1970. **Sample feeder for continuous paper electrophoresis.** A. J. Woiwod and R. Knight (Wellcome Res. Lab., Beckenham, Kent, England). *Lab. Practice*, 1958, **7** (7), 420.—The apparatus (illustrated) consists of an H-shaped glass cell, from one limb of which the liquid to be fed is expelled by means of gas-pressure developed in the other limb by electrolysis of 0·5 *M* K_2SO_4. The liquid is delivered into a trough attached to the electrophoresis apparatus, whence a paper tag, cut but not detached from the main paper, draws it up to the point of attachment. The rate of feed can be varied widely by altering the dimensions of the tag, and optimum conditions for any particular system, obtained by experiment, can be readily reproduced. The rate of feed to the trough is controlled by varying the voltage and current applied to the electrolytic cell. The sample container of the feed vessel, of vol. approx. 5 ml, can be recharged without interrupting the run, but the rate of feed is sensitive to temperature changes, so that the vessel should be protected from draughts and handled as little as possible.
R. E. ESSERY

1971. **Chromatography on paper impregnated with ion-exchange resins. III. The separation of some cation mixtures.** M. Lederer (Inst. du Radium, Paris, France). *J. Chromatography*, 1958, **1** (4), 314–316.—The equation $x\text{pH} = R_M + \text{constant}$, where x is the valency of the cation and $R_M = \log(1/R_F - 1)$ (*cf.* Lederer and Kertes, *Anal. Abstr.*, 1957, **4**, 474), can be used to predict the conditions under which ion-exchange separations can be achieved when only the cation - H^+ equilibrium is concerned, and for choosing a suitable concentration of developing acid. By using paper impregnated with Dowex-50 resin (*cf.* Lederer, *Anal. Abstr.*, 1955, **2**, 1445), and developing with HCl soln. by the ascending technique, the following cation mixtures have been separated, the concentration of developing acid being given in brackets, and the bands being detected with $(NH_4)_2S$ for Cu and Cd, and oxine for the others—Cu - Cd (0·3 to 0·5 *N*); Th - Fe - UO_2^{2+} (0·5 *N*); Th - Ce - Fe (2 *N*); Al - Zr (2 *N*); Ti - Fe - Al (0·5 *N*).
R. E. ESSERY

1972. **Quantitative radial paper chromatography by means of a spectral line photometer.** H. Sulser (Lab. Min. Health, Bern, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1958, **49** (5), 344–347.—Radial paper chromatograms are quantitatively evaluated as follows. A diapositive film is prepared and mounted between two glass plates. The degree of blackening is determined by photometric measurement of successive equal widths of the blackened areas. The extinction values obtained are plotted against the width (mm) scanned and the areas are evaluated planimetrically. The method has been applied to a synthetic mixture of unsaturated acids. Deviations of − 3·3 to + 4·3% were found. The large deviation of − 13% for linoleic acid is accounted for by the poor stability of this particular spot. Fresh chromatograms should be used.
S. BAAR

1973. **Device for the automatic continuous recording of the radioactivity of paper chromatograms.** J. P. Vigne and S. Lissitzky (Lab. des Isotopes, Hôpital Ste Marguerite, Marseille, France). *J. Chromatography*, 1958, **1** (4), 309–313 (in French).—An apparatus is described and illustrated in which the chromatogram is drawn over a slit of adjustable width, situated in front of an end-window β-counter. The current emitted by the counter is passed to an integrator and thence to a recording milliammeter, the cylinder of which can be connected by bevel-gearing with that carrying the chromatogram, so that the speeds of chart and chromatogram are synchronised. The peaks on the chart are evaluated by planimeter. The apparatus is constructed of chromium-plated brass, and is easily demounted for decontamination. The equation $S = V(4T)^2$, where S is slit width, V is chart speed and T the time constant of the integrator, permits the calculation of the best slit-width for a given chart-speed, and, together with the variability of the sensitivity of the integrator over a ratio of 1:10, provides for a wide range of activities. A number of chromatograms can be recorded successively by joining them end to end with Cellophane tape, and an accuracy better than 5% is claimed for the complete sequence of operations, including the chromatography.
R. E. ESSERY

1974. **Gas-phase chromatography.** T. Gramstad. *Tidsskr. Kjemi, Bergv.*, 1958, **18** (6), 81–91.—This is a comprehensive review of the theory and practice of gas chromatography with a description of the construction and use of the apparatus. (15 references.)
N. E.

1975. **Gas analysis apparatus: based on gas-chromatographic and chemical principles.** P. G. Sevenster. *S. Afr. Ind. Chem.*, 1958, **12** (8), 151–158.—The gas mixture is first analysed for CO_2 and O by chemical means. A sample is then introduced by means of a special pipette into a gas-chromatography apparatus which preferably consists of a short carbon column, a silica-gel column, and a long carbon column, CO_2 being used as carrier, and analysis is carried out by means of a nitrometer detector. The nitrometer yields at least as good results as a thermal conductivity cell detector. The activated carbon used in the columns should be chosen with care. The apparatus is cheap, and is quicker to use and more accurate than the Orsat apparatus, particularly for hydrocarbon gases.
A. M. SPRATT

1976. **Use of zeolite in gas chromatography. I. Separation and analysis of mixtures of hydrogen, oxygen, nitrogen, carbon monoxide and methane.** J. Janák, M. Krejčí and H. E. Dubský (Lab. of Gas

Analysis, Acad. Sci., Brno, Czechoslovakia). *Chem. Listy*, 1958, **52** (6), 1099–1107.—The composition, properties and behaviour of calcium zeolite as an adsorbent have been determined by means of differential thermal analysis, X-ray methods and chemical and sorption analysis. The characteristic elution vol. (chromatographic spectra) of H, O, N, methane and CO at different temp. and water content of the adsorbent have been measured. The statistical evaluation of the errors of the methods used in quant. chromatography with thermal-conductivity indication is described. J. ZÝKA

1977. **The sorption properties of dimethyldioctadecylammonium bentonite using gas chromatography.** D. White and C. T. Cowan (Univ., Leeds, England). *Trans. Faraday Soc.*, 1958, **54** (4), 557–561.—The differences in the vapour-phase adsorption of aliphatic and aromatic compounds on dimethyldioctadecylammonium bentonite were investigated chromatographically. Columns with liquid paraffin as the stationary phase and paraffin wax as the adsorbent were also investigated for purposes of comparison. A chromatographic apparatus with a simple hydrogen detector was used; the output was fed into a 0·5-mV recorder. Thermodynamic data were calculated for organo-clay and liquid-paraffin systems. It was found possible to calculate thermodynamic functions from gas - liquid chromatographic data, but difficulties were encountered in the application of these principles to gas - solid chromatography. K. R. C.

1978. **Continuous separation of gases by a new method of counter-current distribution (continuous gas-elution chromatography).** H. Pichler and H. Schulz (Tech. Hochschule, Karlsruhe, Germany). *BrennstChemie.*, 1958, **39** (9–10), 148–153.—The method is based on the distribution of the components of a mixture on two phases in counterflow, one of which is a gas. The second phase is either an inert carrier of large surface area or a solid adsorber. The basic requirement for chromatography—"the uniform percolation of a fluid"—is satisfied with this system. The principles of the method are described with reference to the separation, into two fractions, of similarly volatile materials. The method is demonstrated by the separation of *n*-butane - *trans*-butene (b.p. − 0·5° and + 0·9°) and *trans*-butene - *cis*-butene (b.p. + 0·9° and + 3·7°). The apparatus consists of a column down which the solid phase, aided by a vibrator, moves continuously. The gas mixture is introduced at the centre of the column and the carrier gas at the base. The eluent gas is led off at the top of the column. Chromatograms of the gas mixtures are reproduced. G. P. COOK

1979. **Relationship between peak area ratio and sample composition in gas chromatography.** Yuzi Takayama (Mitsubishi Rayon Co., Kyobashi, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1958, **61** (6), 685–687.—The relationship between the peak area ratio and the composition of the sample was examined for various org. compounds (*e.g.*, methanol, ethanol, *iso*propyl alcohol, $CHCl_3$, benzene, and methyl and ethyl acetates) with Fischer Gulf Partitioner and Super Fractioner GJ. By the use of either thermistor-type or hot filament-type detectors, the peak area ratio is correlated with the volume composition, not with weight or molar composition. The temp., the amount of the sample and the flow rate of the carrier gas have little effect on the peak area ratio. K. SAITO

1980. **Behaviour of katharometers for gas chromatography in carrier gases of low thermal conductivity.** J. Bohemen and J. H. Purnell (Dept. of Phys. Chem., Free School Lane, Cambridge). *J. Appl. Chem.*, 1958, **8** (7), 433–440.—Previous studies of the performance of hot-wire katharometers have not taken into account forced convection. An equation is derived for the rate of heat loss which contains a molar heat capacity term, and which does explain the behaviour of these instruments. Peak distortion and inversion are investigated, and the cross-over temp. is shown to be flow-dependent. J. H. WATON

1981. **Methane-conversion detector for gas chromatography.** A. Zlatkis and J. A. Ridgway (Univ. of Houston, Texas, U.S.A.). *Nature*, 1958, **182**, 130–131.—With hydrogen as carrier gas, compounds emerging from the chromatographic columns are converted into methane and H_2O by passage through a tube of Pyrex glass (24 cm × 5 mm) filled with nickel catalyst and heated at 350° to 450°. Water is removed by a drying column and the methane is measured by a thermal-conductivity cell. Advantages claimed are (*i*) increased sensitivity, as the thermal-conductivity cell is operated at ambient temperature, (*ii*) calibrations for differing conductivity of different compounds are not required, although the carbon number effect must be considered, (*iii*) the method can be applied to other detectors where hydrogen is used as carrier gas, and (*iv*) aq. soln. of alcohols, aldehydes and ketones are easily analysed, as these are readily converted by the catalyst. The catalyst remains active for more than 100 samples. The method has been used for mixtures of C_1 to C_{12} paraffins and olefins, C_5 to C_8 naphthenes, and C_2 to C_5 alcohols, aldehydes and ketones. R. E. ESSERY

Optical

1982. **Semi-quantitative universal method of spectrochemical analysis.** P. V. Mohan and D. L. Fry (General Motors Corp., Warren, Mich., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (3), 90–95.—The method described has been calibrated for the determination of twenty elements in powder samples by the d.c. arc. Lithium carbonate is used as a common matrix. The method is accurate for many concn. ranges to within factors of 0·5 to 1·5 of the amount present. K. A. PROCTOR

1983. **Quantitative spectrochemical methods of broad applicability.** E. K. Jaycox (Bell Telephone Lab., Inc., Murray Hill, N.J., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (3), 87–89.—Techniques are described for d.c. arc spectrochemical analysis of common alloys in which standard samples are used together with synthetic standards. Germanium dioxide is often used as a spectroscopic buffer. The availability of standard samples, their usefulness and limitations are discussed. The precision attainable is comparable with that of other arc methods. K. A. PROCTOR

1984. **Spectrographic analysis by the method of evaporation.** L. V. Lipis. *Zavod. Lab.*, 1958, **24** (6), 736–745.—A review, with 48 references. G. S. SMITH

1985. **Increasing the sensitivity of spectrographic analysis of gases by enrichment in a direct-current discharge.** N. A. Matveeva (A.A. Zhdanov Leningrad State Univ.). *Zavod. Lab.*, 1958, **24** (6),

746–748.—The method is based on the separation that occurs in a capillary discharge tube with d.c. excitation when one component of a mixture of gases has a lower ionisation potential than that of the main gas. It is applied to the determination of argon (2×10^{-5} to 2×10^{-4} %) as an impurity in helium.
G. S. SMITH

1986. **Internally standardised Nujol mull as a method of quantitative infra-red spectroscopy.** K. B. Bradley and W. J. Potts, jun. (Dow Chemical Co., Midland, Mich., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (3), 77–80.—Lead thiocyanate has proved to be generally useful as an internal standard for the quant. application of this technique. In the analysis of known mixtures of phthalic and terephthalic acids the error ranged from + 10% to − 6% of the amount present.
K. A. PROCTOR

1987. **Wide range infra-red absorption cell.** J. U. White and N. L. Alpert (White Development Corp., Stamford, Conn., U.S.A.). *J. Opt. Soc. Amer.*, 1958, **48** (7), 460–462.—A multiple traversal gas absorption cell is described which has been designed for use with Beckman IR-4, -5 and -6 infra-red spectrometers. The vol. of the cell is 6 litres and the cell may be used at pressures up to 10 atmospheres. External adjustment can select path lengths of 10 cm, 1 metre or 10 metres; additional intermediate lengths may also be selected. Excluding window losses, the transmission of the empty cell is 69% at 3 μ and 75% at 14 μ for the 1-metre path, 30% at 3 μ, and 41% at 14 μ for the 10-metre path.
B. S. COOPER

1988. **Simple arrangement for the automatic registration of fluorescence spectra.** J. Jokl (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1958, **52** (7), 1370–1372.—The arrangement described enables spectra to be measured with higher accuracy and speed than when using spectrophotometric methods. The arrangement can be also used for other absorption and emission measurements.
J. ZÝKA

1989. **Use of perchloryl fluoride in flame photometry.** G. E. Schmauch and E. J. Serfass (Chem. Dept., Lehigh Univ., Bethlehem, Pa., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (3), 98–102.—Perchloryl fluoride (ClO_3F) and hydrogen have been used in combination in a modified Beckman burner to give an easily controlled, bluish flame with sufficiently low background for use in flame photometry. An examination of the spectra produced by this flame shows that primarily atomic lines and metal fluoride and metal chloride band-radiation is emitted. A mixture of Mg and Ca excited by this flame produces a spectrum characteristic of both individual elements without any overlapping of the prominent lines or bands. The performance of this gas combination is compared with that of acetylene - oxygen and hydrogen - fluorine.
K. A. PROCTOR

1990. **A remotely operated Hilger Spekker absorptiometer for use with radioactive solutions.** G. P. Kitt and J. H. Moss (A.E.R.E., Harwell, Berks., England). A.E.R.E. Report C/M 356, 1958, 7 pp.—A cell in which colorimetric analyses on samples containing up to 100 mC of $\beta\gamma$ activity have been performed is described and illustrated. The instrument was operated through the wall by means of simple mechanical extensions to the controls. Provision was made for the pipetting, heating, and ultimate disposal of the samples.
G. J. HUNTER

1991. **Apparatus for recording small changes in light transmission in turbid suspensions.** L. Packer (Biochem. Dept., Walter Reed Army Medical Center, Washington 12, D.C., U.S.A.). *J. Opt. Soc. Amer.*, 1958, **48** (7), 503–504.—The apparatus described is a high-gain single-beam recording attachment to a Beckman Model DU monochromator. It is of particular application to the measurement of small changes in the light transmitted by turbid suspensions. The circuit may be used with other types of monochromator.
B. S. COOPER

Thermal

1992. **The study of compounds by means of thermogravimetry.** L. Erdey (Chem. Inst., Tech. Univ., Budapest, Hungary). *Chem. Zvesti*, 1958, **12** (6), 352–365.—Differential thermal analysis, thermogravimetry and derivative thermogravimetry are discussed and examples of practical importance are given.
J. ZÝKA

1993. **Simple automatic, high-temperature thermal analysis apparatus.** S. J. Lloyd and J. R. Murray (A.E.R.E., Harwell, Berks., England). *J. Sci. Instrum.*, 1958, **35** (7), 252–254.—The sample (as small as 0·3 g) is contained in a chamber evacuated to better than 10^{-5} mm and heated by a molybdenum element, the thermal arrests being observed by the differential method. For small samples the specimen is in direct contact with the thermocouple hot junction. Larger samples (20 to 80 g) are contained in crucibles. The apparatus has been used for detecting thermal arrests up to 1550°.
G. SKIRROW

1994. **The air thrust in the bell furnace of the Chevenard thermobalance.** C. Duval (École Nat. Supérieure de Chim., Paris). *Mikrochim. Acta*, 1958, (5), 705–708.—The thrust caused by heated air may be compensated for by mounting an empty crucible in the furnace of the thermobalance. The balance plug should be provided with openings judiciously placed.
D. F. PHILLIPS

Electrical

1995. **Apparatus for high-frequency titrations at a frequency of 500 MHz.** M. Pauček (Biochem. Inst., Agronom. Fac., Brno, Czechoslovakia). *Chem. Listy*, 1958, **52** (7), 1367–1369.—A new apparatus comprises an oscillator, measuring circuit and feeding source and has been tested by neutralisation and precipitation titrations. A detailed description and diagram of the apparatus are given.
J. ZÝKA

1996. **Heterometric titrations.** J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chemie, Prague*, 1958, **10** (7), 539–542.—Principles of heterometric titrations and a survey of all published communications in this field of chemical analysis are given. (44 references.)
J. ZÝKA

1997. **EPR spectrometer of very high sensitivity.** H. Misra (Dept. of Physics, Ravenshaw College, Cuttack 3, India). *Rev. Sci. Instrum.*, 1958, **29** (7), 590–594.—A reflection-cavity-in-magic-T-bridge type EPR spectrometer operating at 3 cm wavelength is described. The instrument will detect about $1{\cdot}1 \times 10^{-11}$ mole of diphenylpicrylhydrazyl in purified benzene (corresponding to $\simeq 10^{-14}$ mole of the crystalline material).
G. SKIRROW

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic.	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{3+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{3-} PO_4^{3-}, etc., for anions.

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